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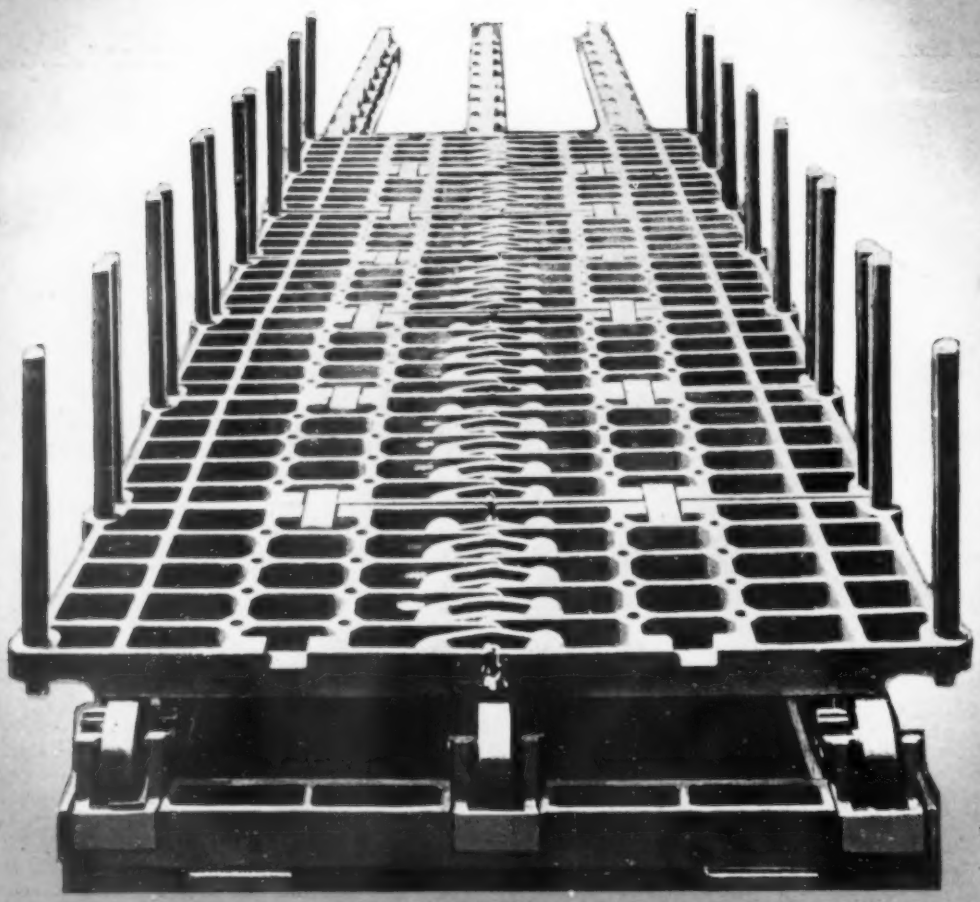

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(INCORPORATING THE METALLURGICAL ENGINEER)

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HALE AND HALE (TIPTON) LTD.

Progress of Subsidiaries—Mr. W. Edgar Hale's Review

THE eleventh annual general meeting of Hale and Hale (Tipton), Ltd., was held Dec. 19th, 1947, at Dudley, Mr. W. Edgar Hale, M.I.Mech.E., M.Inst.F. (the chairman), presiding.

The following is an extract from his circulated review: I venture to suggest that you will agree that the results are quite acceptable, despite the disastrous coal and bad weather stoppage of last winter.

The company's affairs have, during the course of the year, been strengthened from every point of view, which happily has been the case year after year since the very early beginnings of this business, approximately 40 years ago.

PRODUCTION AND SALES

Our production of "fine black-heart malleable iron castings" continues to be accepted as the very finest that is available of its kind. As a raw material to all branches of the engineering trade, and on a value for service basis, it is one of the cheapest commodities available in the engineering world to-day, and its measure of popularity is ever on the increase.

Notwithstanding the difficulties experienced by foundries generally, we have been able to give that excellent service and co-operation with our customers, for which we have a unique reputation in our particular sphere. The quality of our product has been maintained at a very high standard, and our metallurgical staff are constantly engaged in research and experimental work for the improvement of castings, thus keeping "Hales' fine black-heart malleable" to the forefront.

We have an order book which is still heavily loaded, and in some sections have on hand in the region of two years' work. The major portion of our orders are connected directly or indirectly with export. Most of the castings we are making go into the production of light and heavy commercial vehicles, heavy industrial switchgear, agricultural machines of every description, railway rolling stock, permanent way material, signal gear, all types of heavy industrial plant and mining equipment. Unfortunately we have been unable to satisfy the great demand for our product, but we view the coming year with renewed optimism, and are placing our sales targets higher than before. We are confident that our contribution to export and priority industries will be very substantial indeed.

We are compelled, of course, to direct our attention into those channels which we are called upon from time to time by various Government Departments, with whom, I am pleased to say, we work in very excellent harmony. However, I am sure that while all industrialists feel that essential controls are absolutely necessary in the present circumstances, they look forward with continued and ever-growing hope to the day when so much time need no longer be spent by valuable executives in "working controls."

Whatever the future has in store, our company will fare as well as most, and, maybe, better than the majority, because our affairs are in good order, well controlled and managed in every respect, and supported by the good will and co-operation between the personnel and the management. We can, therefore, look forward, subject only to some unforeseen circumstances arising, to steady and successful years in the future.

SUBSIDIARIES

Chatwins, Ltd., is continuing to make very marked and steady progress in its production of "Sunbeam" Household Grates, Ranges, and Domestic Heating Boilers, and I do not think that I have ever known a manufacturing enterprise which had been reduced to a low level, reflect quicker to new and better ideas of management than this one. It is unrecognisable as compared with when I purchased it on behalf of our company.

There is a high standard of efficiency and good will between all parties at this establishment, resulting in an improved standard of product in an ever-increasing volume. The order book is in very good shape, and the company's appliances are recognised by customers as of very high standard, first-class design, quality, and finish, being more than comparable with any on the market. Many of the largest municipal authorities engaged upon house building schemes have specified with considerable enthusiasm our products.

During the course of the coming year we hope to have on the market some very attractive new appliances for both cooking and heating that will be quite revolutionary, and we predict real "business spinners."

A process of careful cultivation is being applied to export markets and the outlook is very encouraging.

J. and J. Whitehouse (Tipton), Ltd., too, can be referred to in precisely the same terms as the Chatwin subsidiary, except that it is engaged upon the production of cast iron hollow-ware almost exclusively for tropical countries, and in its own particular sphere is making a very useful contribution to our national exports.

THE ACCOUNTS

The trading profit amounts to approximately £25,000. From this figure has to be deducted depreciation, etc., leaving a net profit for the year of roughly £19,000, to which must be added the amount brought forward from the previous year of approximately £14,000. Thus we had at our disposal a total of £33,000.

Your directors have given careful consideration to what they feel should be a desirable final dividend for the year on the Ordinary shares, and while recognising the Ordinary shareholders have never been generously dealt with, your directors are of the opinion that they are not justified in moving away from their established policy of having a definite bias in favour of a conservative outlay in this connection. The average dividend over the years is not a high one, but during those years the company has been steadily built up and has justified its very important function of maintaining sound security and permanent employment for all who are dependent upon it for their livelihood.

It has, therefore, been recommended to pay the same dividend as last year—namely, 15 per cent.—making a total of 20 per cent. for the year.

The report and accounts were adopted.

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THE BRITISH JOURNAL OF METALS.

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JANUARY 1948 Vol. XXXVII No. 219

Oxygen in Steelworks

SINCE our previous reference[1] to the above subject further valuable contributions have been made to a discussion of rapidly growing importance, including two papers read at the Zurich meeting of the Iron and Steel Institute, and an editorial conference of the Linde Air Co. in the United States. The first of the two papers was that by Dr. R. Durrer, on oxygen-enriched blast,[2] who reached the conclusion that, although much more preparatory work is needed to overcome many technical and economic problems, these latter are not fundamental. Basically the idea is sound and will eventually be fully realised. In the second paper referred to, Dr. Thring discussed the possibilities of the extended use of oxygen in the British iron and steel industry[3]; he too, concluded that recent developments in methods of manufacture of oxygen in the two purity stages 80–100% and 40–60%, make its use in fuel combustion in steel manufacture of considerable interest.

At a recent conference, the Vice-President of the Linde Air Co., and others, gave oral and written information on some of the latest results achieved in the use of oxygen in blast furnace practice.[4] They described improved methods of producing very cheap oxygen for this purpose on a tonnage basis. This Company has been producing gaseous oxygen in a pilot plant at East Chicago since December, 1946, as a result of experimental work begun in 1932 and based on the Linde–Frankl cycle. They claim to produce 96% oxygen under 3 lb. pressure for less than 6 dol. per ton, however, all difficulties are not yet overcome. Capacity of the pilot plant is 4·8 mill. cu. ft. (200 tons) per day of 90% grade from a single fractional distillation unit.

As with all oxygen plants there are three fundamental phases: (1) purification of air; (2) partial liquefaction of air; and (3) separation of partially liquefied air into oxygen and nitrogen by distillation. Since it is immediately available in their works air for the new plant is already under pressure of 80 lbs./sq. in. from the second compression stage in the production of liquid oxygen. The quantity of air passing through the gaseous plant is so large that the usual L.F. (Linde–Frankl) chemical purification is not possible; water vapour and carbon-dioxide are removed by regenerators working under pressure, with very large heat exchange surfaces, e.g., corrugated aluminium ribbon in spiral form packed in the tower so that 1 cu. ft. of packing gives 2,000 sq. ft. of surface. Four regenerators are required: two working alternately on air and nitrogen and two alternately on air and oxygen. Although air at present used is compressed by reciprocating units centrifugal compressors will be used in future, as more efficient. The gaseous oxygen from regenerators contains small amounts of water vapour and carbon dioxide which in many industrial uses is not objectionable but where pure gas (99·5%) is required heat exchangers can be substituted for air-oxygen regenerators wherein the oxygen is not exposed to the moisture or carbon dioxide removed from the impure air.

The company was mainly interested in oxygen for steel making, and in quoting a price of 6 dol. per ton or less certain conditions must be fulfilled. In the first place the plant must operate on a load as nearly constant as possible. For some months past a careful study had been made of the probable oxygen demand in steel plant, of 99·5% grade for scarfing, cutting and welding etc. and a curve representing this demand shows extreme variations. A similar demand curve was drawn for oxygen of lower grade for use in open hearth furnaces, e.g., for melting down scrap and decarburising; and this, too, showed similar variations. To meet the peak loads plant of excessive size and capacity would be needed.

Then there is the problem of periodical close-down for thaw-out and minor repairs and sometimes for major repairs, including the removal of accumulated impurities such as water vapour and carbon dioxide. For both peak loads and close-downs the question of storage therefore arises, with supplementary queries as to high or low pressure storage and the matter of stand-by equipment.

Much work has been done in the U.S.A. on use of oxygen in open hearth and electric furnace, and liquid gas at 36 dol. per ton has been supplied for the purpose to a number of steel companies. Tests have been made at 24 mills and records of 3,000 heat tests have been secured. Little has so far been done with blast furnaces. Oxygen has been used to shorten both cycles in open hearth. The melting down period has been shortened by four different methods: (*a*) enrichment of air by passing oxygen through end burners or through auxiliary equipment placed near; (*b*) scrap melting by directing oxygen on to preheated scrap; (*c*) use in auxiliary burners to accelerate melting down: and (*d*) enrichment of air by injecting oxygen into intake to checkers. One way of introducing oxygen was through the main burners.

These methods shorten melting down by raising the rate of burning fuel, increasing the flame temperature in contact with scrap, also increasing and directing radiation, and by actually burning iron in the scrap. The refining period has also been shortened by introducing additional oxygen into the molten bath, resulting in rapid reduction of carbon content, and agitation of the slag/metal interface.

Selection of most suitable method of use depends on

1 *Journ. I. S. Inst.*, 1947, pp. 55–56.

2 *Journ. I. S. Inst.*, 1947, **156**, pt. 2, 253–260.

3 *Journ. I. S. Inst.*, 1947, **156**, pt. 2, 285–291.

4 *Chem. Eng. Progress*, 1947, **43**, 16–17 and 28; also *Blast Furnace and Steel Plant*, 1947, **35**, 813–6.

percentage of cold charge facilities for charging, type of steel produced, and furnace conditions. Increased productivity per furnace can be achieved if the various necessary facilities and capacities—tapping, rolling mill, etc.—are provided accordingly; or alternatively the same amount of steel can be produced from smaller number of furnaces, e.g., by closing down those of lower efficiency. Scrap melting down with oxygen will be the more advantageous of course, as scrap percentages are high, i.e., at least 35% of the total.

About 3·5–4·5 mill. B.t.u. are required per ton of steel. In shops using 100% cold charge, heat input rises to as much as 5·5 mill. B.t.u. per ton or more. Approximately 20–25% of this heat enters the steel and the rest is lost to the furnace, waste heat boilers, and stack. The usual fuel (oil) requirements per heat for a ton of steel are approximately 20–30 galls., based on 150,000 B.t.u./gall. Complete combustion of 20–30 gall. of Bunker C oil, heavy fuel oil, or tar requires 6,500–10,000 cu. ft. oxygen which may be supplied entirely from air or oxygen-enriched air; 20–30% of the total required may be provided through main burners.

It was found that oxygen increased radiation from the flame by about 10% throughout the greater part of flame, and theoretical considerations indicate increase in the average flame temperature of about 200–400° F. Results of 728 heats showed that oxygen gave 10–25% reduction in charge-to-tap time, oxygen consumption being 400–750 cu. ft./ton per heat. Under the best conditions these results can be improved. By way of example capacities of 125 ton open hearth were increased 32% with 337 cu. ft./ton.

In their large Tonawanda laboratories, the company has developed oxygen end burners and also jets capable of delivering high velocity gas right into the centre of the open hearth without being immersed in the bath. These oxygen jet experiments are not yet completed, but using oxygen at 75–100 lbs./sq. in. and flow rate of 30,000 cu. ft./hr. on a charge comprising 60% cold scrap, 25% reduction in charge-to-tap time resulted. This is a new technique in which the jets are inserted through holes in the furnace and continually moved to cause high velocity gas to impinge on unmelted portions of the charge at a working distance of 10–30 ft. Six heats were made in a 175-ton furnace equipped with burners in the roof. Average saving in time was 1·6 hr. with consumption of 200 cu. ft./ton. Similar savings are also recorded for decarburisation. Most of the oxygen in this work was obtained from the liquid product as obtained in the Linde Driox systems.

With reference to the effect on refractories, wear is not increased if the right type is used and necessary precautions taken. Chrome-magnesite refractories are being tried at temperatures up to about 3,000° F. Roof life in some shops was shortened, mainly due to violent splashing, and can be remedied by increase in roof height. Purity of oxygen need not be above 90%, or may be even down to 75%. Work is also being done on compressed air. The results so far achieved must be regarded as tentative and will need further study and full scale experiments, but it seems apparent that the greater use of oxygen in iron and steel plants will increase production. It is noteworthy, for instance, that oxygen, added directly to the bath in open-hearth furnaces, for controlling the bath temperature and for shortening the refining period has become standard practice in several plants in the United States making low-carbon steels.[5]

5 "Practical Experience with the Use of Oxygen in Steelmaking" by G. V. Slottman, *Jour. I. S. Inst.*, 1947, **157**, pt. 3, 331–336.

Steel Consumers' Census

THE Government has decided to resume a census of receipts, consumption and stocks of steel by consumers, which was in operation during the war, but was discontinued in 1945 on the termination of hostilities. With this object a form will have been received by some 18,000 steel-using firms during the last week or so, on which are to be entered receipts and consumption of steel, by the particular firm concerned, during the fourth quarter of 1947, and of stocks as at December 31, 1947. It is considered that the changed pattern of demand by consuming interests and the forthcoming revision of the Iron and Steel Distribution Scheme, make the resumption necessary and a matter of urgency.

The completion of the form obviously involves additional work on the part of consumers, but the importance of ensuring a proper distribution of the available supplies of steel outweighs any minor inconvenience involved. The form has been prepared in what is regarded as the simplest possible way; only six figures are asked for and it is stated that the prompt despatch of the return by industry will greatly assist in bringing into force the improved scheme of distribution.

Many firms with a diversity of interests will find the filling of this form more difficult than it appears, especially in view of the fact that the revised distribution scheme will involve adjustments so that more steel can be allocated for the production of mining and agricultural machinery, engineering and various types of vehicles, consumer goods, railway reconstruction and maintenance and new power stations.

Some modification in allocation has become necessary because the target of 14 million tons set for 1948 is not enough to meet requirements. For essential purposes alone, Britain could use 10½ million tons of finished steel, but it is estimated that only 9½ million tons will be available if the target is achieved. It should be kept in mind that the Government has decided to export a million tons, since this is considered essential to the conclusion of many bilateral trade negotiations and to enable Britain to play her part in world reconstruction. It is likely that some 500,000 tons of steel will be imported—not as much as we could effectively use, but a welcome addition to the amount which should become available.

The estimated total of 9½ million tons to meet home demands is considerably in excess of that available last year, when a little over 8 million tons were allocated. It would seem, therefore, that very few industrial concerns will get less steel than last year, while many will get more. Much depends upon the iron and steel industry in reaching the output target. There seems every confidence that it will be exceeded, provided fuel and scrap are available, and, if so, it will exceed in tonnage any previous annual production. There is no doubt that the industry will do everything possible to achieve the target and thus ensure consumers of their allocation.

The Control of Gas Content During the Melting of Phosphor Bronzes, Gun Metals and Leaded Bronzes

By W. T. Pell-Walpole, D.Sc., A.I.M.

Information is given on the quantitative control of degassing phosphor bronzes, gun metals and leaded bronzes, by use of oxidising fluxes, as a result of a detailed investigation on the effects of (a) the CuO content of the flux and (b) the amounts of gas and of phosphorus or zinc in the charge, on the efficiency of degassing and on the oxidation losses.

IT is well established that binary tin-bronzes containing only such small amounts of phosphorus or zinc as may result from deoxidation treatment, can be effectively degassed by melting under oxidising conditions, preferably under a fluid oxidising flux, followed by deoxidation.[1-3] Most of the bronzes used industrially, however, contain much larger amounts of phosphorus (up to 2%), zinc (up to 5%), or lead (up to 20%). If such alloys are prepared from virgin metals or from charges of binary bronze, they can be effectively degassed by the same procedures used for binary bronzes, provided the oxidisable elements are not added until after the oxidising flux has been removed from the melt (Table I).

In normal industrial practice the charges for ternary bronzes are commonly composed of either a mixture of scrap and virgin metals, or of ingoted metal of the required composition. These conditions, in which the oxidisable element is already present in the charge, render degassing by oxidation less straightforward, since the phosphorus (or zinc) present in the charge can compete with the hydrogen for combination with the available oxygen of the flux. It is to be expected therefore that more strongly oxidising treatments will be required to degas this type of alloy, and that there will be an appreciable melting loss of the zinc or phosphorus present, due to oxidation of these elements and solution in the flux of the oxides so formed.

Baker and his colleagues[4] found that oxidation treatments which were quite satisfactory for degassing tin-bronzes of low zinc or phosphorus content were relatively useless with gassy charges of high-phosphorus bronzes or of gun-metals. They considered that this was due to the fact that degassing by oxidation can be effective only if the oxygen can be introduced into solution in the molten metal, and showed that oxygen is practically insoluble in bronze containing more than a trace of phosphorus or zinc. They contended that these elements would have to be removed completely by oxidation before degassing could be effected by oxidation. They therefore abandoned oxidation treatments for these alloys and developed alternative methods embodying the principle of scavenging. On the other hand, the present author and others[5, 6] had found that in practice an oxidising flux containing cupric oxide, sea-sand and fused borax gave quite satisfactory degassing (Table II) with scrap charges of phosphor bronzes or gun metals, provided that the CuO content was not less than 33% and that the amount of flux used was about 3% of the charge by weight. The losses of phosphorus and zinc involved were quite moderate (Table II) and could be replaced by further additions after removal of the flux from the metal. This flux process ensures a much higher proportion of available oxygen (in the form of CuO) than was used by Baker in his experiments; the flux also operates in a protective and degassing capacity from the commencement of melting, not merely for a short time, after melting is complete. These differences probably

1 Pell-Walpole, W. T., *J. Inst. Metals*, 1940, **70**, 127.
2 Pell-Walpole, W. T., *J. Inst. Metals*, 1941, **71**, 37.
3 Pell-Walpole, W. T., *J. Inst. Metals*, 1941, **71**, 267.
4 Baker, W. A., and Child, F. C., *J. Inst. Metals*, 1944, **70**, 349.
5 Joint Discussion on Bronze Melting. *J. Inst. Metals*, 1944, **70**, 557.
6 Pell-Walpole, W. T., *Metallurgia*, 1946, Vol. 34 (200), 85 (201), 155.

TABLE I.—THE DENSITY AND PROPERTIES OF CHILL-CAST 2-IN. DIAMETER INGOTS OF PHOSPHOR BRONZES AND GUN METALS, PREPARED FROM VIRGIN METALS UNDER OXIDISING FLUXES.

Alloy Type	Analysis: Tin %	Phos. %	Zinc %	Lead %	Flux*	Pouring Temp. °C.	Pouring rate lb./min.	Bulk dens. gm./cc.	Properties of upper test piece: dens. gm./cc.	U.T.S. tons/in.	El % on 2 in.	Properties of lower test piece: dens. gm./cc.	U.T.S. tons/in.	El % on 2 in.
2B8	10·4	0·77	—	—	2% SB½	1250	8	8·75	8·79	27·2	33	8·78	26·8	25
	10·8	0·81	—	—	4% —	1250	8	8·75	8·78	27·5	24	8·77	26·4	17
	10·6	0·76	—	—	8% —	1250	8	8·75	8·79	28·0	24	8·77	27·8	27
	10·2	0·66	—	—	3% SB½	1250	8	8·75	8·78	27·5	21	8·71	26·2	18
	10·1	0·82	—	—	3% SB½	1250	8	8·75	8·80	26·6	24	8·73	26·5	15
High phosphor bronze	9·3	1·9	—	—	3% SB½	1200	7	8·65	8·65	30·6	15	8·53	28·6	10
Phosphor bronze	8·2	0·35	—	—	3% —	1200	10	8·85	8·85	24·0	38	8·83	25·2	48
Medium phosphor	9·8	0·25	—	—	3% —	1250	10	8·85	8·85	25·4	54	8·86	22·1	17
	10·0	0·50	—	—	3% —	1250	8	8·84	8·85	26·8	36	8·80	25·0	21
Gear bronze	13·0	0·20	—	—	3% —	1250	8	8·86	8·82	22·8	5	8·82	22·7	5
Admiralty	10·2	0·04	2·5	—	3% SB½	1200	10	8·86	—	19·3	9	—	—	—
Gun metal	10·2	0·05	2·1	—	3% SB½	1200	10	8·87	8·86	22·0	18	8·86	22·5	16
	10·6	0·10	1·9	—	3% SBO	1200	10	8·83	8·78	16·7	7	8·79	18·7	7
	10·1	0·06	1·9	—	3% SBO	1200	10	8·83	8·81	17·9	8	8·80	17·6	8
High zinc gun metal	7·2	0·08	5·1	—	3% SB½	1200	10	8·86	8·84	23·8	3	8·84	19·0	1
Gun metal 88/8/4	8·6	0·19	3·9	—	3% SB½	1200	10	8·82	8·81	20·2	16	8·81	21·0	18
Leaded gun metal 85/5/5/5	5·3	0·07	4·2	4·9	3% —	1200	9	8·88	8·87	19·0	33	8·87	19·0	32
Leaded phosphor bronze	7·6	0·42	1·3	3·4	3% —	1200	8	8·84	—	25·3	28	—	23·9	16
Leaded bronze 10%	9·7	0·05	—	8·4	3% —	1200	9	8·97	8·97	19·4	16	8·94	18·4	13
Leaded bronze 20%	8·3	0·14	—	20·1	3% —	1200	12	9·17	9·17	16·8	22	9·12	15·2	19

* The fraction denotes the CuO content of flux. Other constituents equal parts of sea-sand and fused borax.

TABLE II.—Properties of Chill-cast Ingots of Gun Metals and Phosphor Bronzes Prepared from Scrap Charges under Oxidising Fluxes.

Scrap Charged	Analysis of treated ingots: Tin %	Phos. %	Zn %	Pb %	Flux used*	Additions	Bulk dens.	Porosity %	Properties of upper test piece: Dns. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
2B8, 10% tin	10·4	0·78	—	—	3% SB½	0·2%P.	8·80	—	8·81	28·9	43	8·79	27·6	29
	10·1	0·44	—	—	3% —	—	8·31	—	8·87	24·2	23	8·83	25·4	32
0·65% phosphor	10·2	0·79	—	—	3% SBO	0·1%P.	8·65	1·1	8·75	26·0	16	8·61	22·0	11
	10·0	0·92	—	—	3% —	0·3%P.	8·62	1·2	8·71	25·6	16	8·60	23·6	13
	11·3	0·99	—	—	6% —	0·3%P.	8·61	1·2	8·64	25·4	11	8·64	23·3	7
	11·3	0·65	—	—	3% —	0·1%P.	8·71	0·7	8·74	24·5	17	8·69	21·0	6
	11·1	0·94	—	—	2% SB½	0·3%P.	8·63	1·1	8·61	23·8	9	8·56	20·4	2
	10·6	0·83	—	—	3% —	0·3%P.	8·72	0·2	8·70	26·2	18	8·69	24·2	9
	10·5	0·85	—	—	4% —	0·3%P.	8·71	0·3	8·72	26·7	25	8·72	24·8	15
	10·4	0·88	—	—	8% —	0·3%P.	8·74	—	8·75	26·1	18	8·73	24·2	14
	10·3	0·86	—	—	3% SB½	0·3%P.	8·71	0·2	8·75	25·0	16	8·67	25·2	10
	9·8	0·84	—	—	3% SB½	0·3%P.	8·71	0·2	8·73	27·0	29	8·69	25·2	17
Gun metal—10% tin, 2·5% Zn	10·6	0·10	1·89	—	3% SB½	½% Zn	8·84	—	8·83	19·8	11	8·82	20·8	13
	10·3	0·05	2·10	—	3% SB½	—	8·83	0·1	8·83	19·9	14	8·82	18·8	11
Gun metal—8% tin, 4% Zn ..	8·6	0·09	4·6	—	3% SB½	—	8·84	—	8·84	22·0	18	8·76	20·9	16
Gun Metal—88/7/5	7·0	—	4·7	1·9	3% —	—	8·81	—	8·81	18·8	19	8·78	20·2	22
Leaded gun metal 85/5/5/5 ..	5·3	0·19	4·6	5·1	3% —	—	8·84	0·2	8·85	20·4	38	8·85	19·7	32

* Fraction denotes CuO content of flux. Remainder consists of equal parts by weight of sea-sand and fused borax.

account for the apparent disagreement with Baker's findings, but the mechanism of degassing in this case must also be different from that postulated by Baker. It is suggested that the degassing reaction occurs at the flux-metal interface, simultaneously with the oxidation of either phosphorus or zinc, thus :—

(1) $2H + CuO \rightleftharpoons H_2O + Cu$.

(2) $P + 3CuO \rightleftharpoons CuPO_3 + 2Cu$.

(3) $Zn + CuO \rightleftharpoons ZnO + Cu$.

In a closed system the oxidation of phosphorus or zinc might tend to take place in preference to the oxidation of hydrogen, but in practice the H_2O can escape by bubbling through the flux while the zinc oxide and the copper or tin phosphates formed dissolve in the flux and thus tend to remain in equilibrium with the molten metal. The removal of hydrogen therefore tends to proceed to completion while the other reactions tend to reach an equilibrium condition.

In order to obtain more information on the quantitative control of degassing these alloys by use of oxidising fluxes, a detailed investigation was carried out on the effects of (*a*) the CuO content of the flux, and (*b*) the amounts of gas and of phosphorus or zinc in the charge, on the efficiency of degassing and on the oxidation losses.

Degassing 2B8 Bronze (10–11% Tin, 0·7% Phosphorus)

Gassy Scrap Charges.—The theoretical considerations in the introduction suggest that the gas content of the treated bronze will depend on (*a*) its initial gas content before melting, (*b*) its zinc or phosphorus content, (*c*) the oxygen content of the flux.

Preliminary tests were made to determine the effect of the CuO content of the flux on the degassing of gassy charges of scrap 2B8 bronze (10–11% tin, 0·6–0·8% phosphorus). The fluidity of the flux, which may affect the speed of the reactions at the flux-metal interface was kept approximately constant by maintaining a constant sand-borax ratio in all fluxes tested. It was found that variation of the CuO content (up to 50%) had little effect on the fluidity. Since density values were to be used as a measure of the gas contents of the treated bronze melts, it was necessary to take precautions against variations in composition, and to avoid the occurrence of other types of porosity in comparative ingots.

Charges of 12 lb. each, of gassy 2B8 scrap bronze (average density 8·60 gms./cc.) were melted under covers (3% of the charge by weight) consisting of $\frac{1}{12}$ to $\frac{1}{3}$ CuO by weight respectively, with the balance of equal parts of sea-sand and fused borax. The flux was charged with the scrap and after melting was left on the metal until the temperature was 1,300° C. The flux was then thickened with dried sand and removed, and small additions of phosphorus (0·1—0·3%) were made to compensate for oxidation losses as determined by previous experience. The melts were poured into 2 in. diameter thin-walled chill moulds, at 1,200° C., 8 lbs./minute, conditions known to ensure freedom from shrinkage porosity, and to produce ingots from gas-free melts having a density of 8·76 ± 0·02 gms./cc.[6] The ingots were inspected for exudation, and density was determined on the whole ingot. The porosity per cent. was calculated by comparison with an "ideal" density for each composition concerned, i.e., the density obtained for a gas-free virgin melt. Two test-pieces were then made from the upper and lower halves respectively. All ingots were analysed for phosphorus contents and tin was determined in a few. The results of these tests are summarised in Table III.

TABLE III.—Properties of 2B8 Ingots Prepared from Gassy Scrap Charges under Various Oxidising Fluxes, in a Crucible Used Previously with a Strongly Oxidising Flux.

Flux cover*	Addition % Phos.	Analyses of: Phos. %	Tin %	Appearance of ingot tip	Bulk dens. gm./cc.	Ideal dens.	Porosity %	Properties of upper test piece: Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
SBO	0·1	0·77	10·6	Trace of exudation	8·72	8·76	0·45	8·76	24·8	16	8·68	21·4	7
SBO	0·3	0·84	10·6	ditto	8·68	8·74	0·70	8·71	25·2	17	8·63	23·4	12
SB$\frac{1}{12}$	0·1	0·69	—	ditto	8·73	8·78	0·60	8·74	27·6	36	8·70	23·3	17
SB$\frac{1}{12}$	0·1	0·66	—	ditto	8·73	8·78	0·60	8·77	26·1	38	8·72	25·6	30
SB$\frac{1}{4}$	0·1	0·65	—	None	8·74	8·78	0·45	8·77	25·8	37	8·73	23·2	18
SB$\frac{1}{4}$	0·3	0·76	10·4	—	7·75	8·76	0·80	8·78	24·6	22	8·75	24·0	21
SB$\frac{1}{4}$	0·3	0·64	10·1	—	8·75	8·78	0·35	8·80	26·6	17	8·78	25·8	21
SB$\frac{1}{4}$	0·1	0·51	—	—	8·75	8·80	0·60	8·78	25·6	39	8·71	23·9	23

* Fraction denotes CuO content. Other constituents equal parts by weight of sea-sand and fused borax.

Fig. 1a.—Ingot-head from melt of gassy 2B8 scrap, melted under sodium chloride cover in a clean pot.

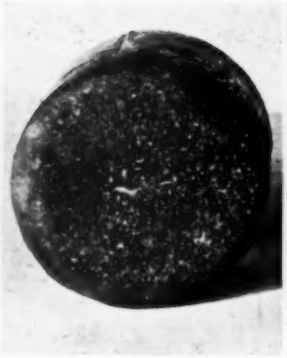


Fig. 1b.—As Fig. 1a, melted under sodium chloride cover in pot impregnated with sand-borax flux.

Fig. 1c.—As Fig. 1a, melted under sodium chloride cover in pot impregnated with strongly oxidising copper-oxide-sand-borax flux.

melting. The ingot melted in the sand-borax pot had slight exudations (Fig. 1b) and much less porosity, indicating that the flux present in the crucible walls had prevented any further gas pick-up, though no positive degassing had occurred. The ingot melted in the pot impregnated with the oxidising flux showed no exudations (Fig. 1c) and only 1% more porosity than a degassed melt made from virgin metals, i.e., a considerable degree of degassing had been effected by the oxidising flux in the crucible walls.

These tests show the important action of the flux carried in the walls of the crucible, and indicate that an oxidising flux process cannot be tested effectively until the crucible has become thoroughly impregnated with the particular flux concerned. This is best effected by making a melt of flux alone in each new crucible before using it for bronze melting. (Sand-borax-CuO fluxes have no appreciable attack on the crucible walls).

The Effect of Oxidising Flux Present in the Walls of the Crucible

The results of the above series of tests showed no evidence of gas porosity except in the ingot made with the sand-borax flux containing no CuO. Throughout the series of oxidising fluxes there was no appreciable variation of density or properties with variations in the CuO content of the flux, the values being almost equal to those obtained with degassed virgin ingots. It seemed that the rich oxidising flux ($\frac{1}{3}$ CuO) with which the crucible had become impregnated from previous use supplied sufficient oxygen (together with that from any of the fluxes used in the present tests) to effect fairly complete degassing of the charge. No further gas pick-up could occur during melting due to the protective action of this flux impregnated in the walls of the pot and the fluid cover on the surface of the melt provided by the flux under test.

In order to determine more fully the effect of flux present in the walls of the crucible three further comparative tests were made, using in each case a non-oxidising cover (4–6% NaCl). In the first test a clean crucible was used, in the second a crucible used previously with a flux consisting of sand and borax only, in the third a crucible used previously with the rich oxidising flux ($\frac{1}{3}$ CuO, $\frac{1}{3}$ Borax, $\frac{1}{3}$ sea-sand) as before. The charge was gassy 2B8 scrap as in the previous tests. The ingot heads are illustrated in Figs. 1a–c, and the test results are given in Table IV.

The ingot prepared from the clean crucible showed severe exudations (Fig. 1a) and had a very low density (8·37 gm./cc.) showing that the charge had absorbed additional gas (through the crucible walls) during

The Effect of the CuO Content of the Flux

Tests on the effect of the CuO content of the flux were repeated, the fluxes being tested in order of ascending CuO content, commencing with a crucible which had been used previously with sand-borax flux only. The charges were gassy 2B8 scrap.

The results, summarised in Table V, show that the sand-borax flux gave an ingot having the same density as one prepared under the NaCl cover when used with a similar pot impregnated with sand-borax flux, i.e., there was no appreciable degassing action. As the CuO content of the flux was increased, the porosity of the ingots decreased regularly (Fig. 2) to 0·3% (compared with that of a degassed virgin ingot) when the flux contained $\frac{1}{6}$ CuO, and to 0·1% with the flux containing $\frac{1}{3}$ CuO. Maximum mechanical properties were obtained only with the richest flux. The flux containing $\frac{1}{6}$ CuO leaves traces of fissure gas porosity, which have little effect on density but prevent maximum mechanical properties being realised. Similar tests on the effect of melting without flux but with an oxidising-flame, included in Table V and Fig. 2, show that these conditions produce only partial degassing, the effect being equal to melting with a flux containing less than $\frac{1}{6}$ CuO.

TABLE IV.—The Effect of Crucible Condition on the Properties of Ingots Made from Gassy 2B8 Scrap Under a Cover of 4% NaCl.

Condition of Crucible Used	Analyses of treated ingot		Ideal dens. gm/cc.	Obsd. bulk Dens. gm./cc.	Porosity %	Properties of copper test piece			Properties of lower test piece			Appearance of ingot head
	Tin %*	Phos. %				Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	
Clean, freshly annealed	10·5	0·73	8·76	8·37	4·5	8·28	13·8	3	8·34	14·4	3	Severe exudations (Fig. 1a)
Impregnated with sand/borax flux from previous use	10·5	0·74	8·76	8·60	1·8	8·62	22·0	9	8·58	22·1	11	Slight exudations (Fig. 1b)
Impregnated with sand,/Borax,/CuO_2 oxidising flux ..	10·5	0·75	8·76	8·68	0·9	8·73	24·3	14	8·68	23·0	13	No exudations (Fig. 1c)

* Normal.

Fig. 2.—Effect of CuO content of flux on the properties of bronze ingots prepared from gassy scrap charges.

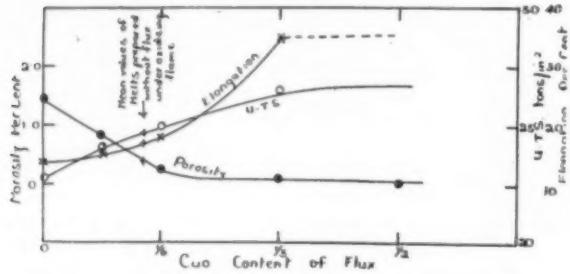
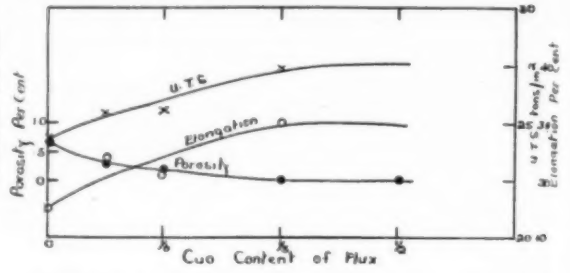


Fig. 3.—The effect of CuO content of flux on the properties of 2B8 ingots prepared from ingoted charges of low initial gas content.



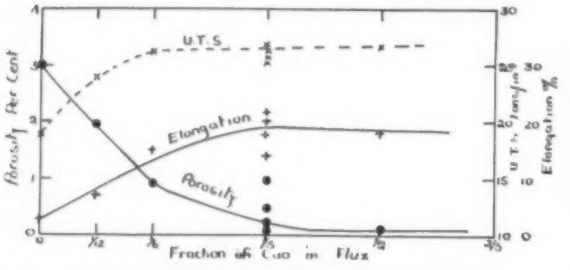


Fig. 4.—Effect of CuO content of flux on ingots of high phosphorus bronze prepared from scrap charges.

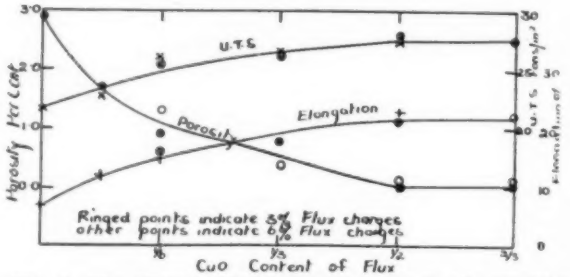


Fig. 6. The effect of CuO content of flux on the porosity and properties of ingots of high phosphorus bronze (1·5%) prepared from gassed virgin charges.

A further confirmatory test of the efficiency of CuO-sand-borax flux for degassing gassy charges of 2B8 scrap was made by melting a charge consisting entirely of oily swarf from sand-castings under the flux containing $\frac{1}{3}$ CuO. The ingot obtained showed no signs of exudations, had a density of 8·75, and tensile strength 26 tons/in.² with 16% extension. When melted without the oxidising flux cover this material had given ingots with severe exudations, internal gas-holes and very poor mechanical properties.

Charges of Ingoted 2B8 Bronze of Low Initial Gas Content

A similar series of tests was made on normal ingoted 2B8 bronze, i.e., a charge of initial low gas content. The results (Table VI and Fig. 3) show that with this type of charge even a protective cover (sand-borax) gives reasonably gas-free ingots, while a weaker oxidising flux ($SB\frac{1}{6}$) (CuO, 1 part, borax, $2\frac{1}{2}$, sand, $2\frac{1}{2}$) gives practically complete freedom from porosity though some

TABLE V.—Degassing Gassy Scrap Charges of 2B8 Bronze (10·5% Tin, 0·7% Phos.)

*Flux cover	Phos. added %	Analyses of Ingot: Tin %†	Analyses of Ingot: Phos. %	Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Appearance of ingot head	Properties of upper test piece: Dens. gm./cc.	Properties of upper test piece: U.T.S. ton/in.²	Properties of upper test piece: El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	Properties of lower test piece: U.T.S. ton/in.²	Properties of lower test piece: El% on 2 in.
3% SBO	0·1	10·5	0·74	8·76	8·64	1·4	Some exudation	8·67	23·6	13	8·56	22·4	14
6% SBO	0·1	10·5	0·73	8·76	8·64	1·4	ditto	8·58	22·4	14	8·67	22·4	14
3% $SB\frac{1}{12}$	0·1	10·5	0·68	8·77	8·70	0·8	Trace exudation	8·74	24·3	13	8·72	23·7	16
3% $SB\frac{1}{4}$	0·1	10·5	0·67	8·78	8·76	0·23	Clean	8·78	24·4	19	8·74	25·3	16
3% $SB\frac{1}{3}$	0·1	10·5	0·66	8·78	8·77	0·11	—	8·77	26·8	39	8·75	26·2	30
No flux oxidising flame	0·1	10·5	0·75	8·76	8·73	0·33	Trace exudation	8·77	25·3	14	8·73	24·1	11
No flux oxidising flame	0·1	10·5	0·84	8·74	8·70	0·46	—	8·70	26·0	19	8·69	24·2	13

* Fraction denotes CuO content of flux. Other constituents are equal parts by weight of sea-sand and fused borax.
† Nominal.

TABLE VI.—Degassing Ingoted Charges of 2B8 Bronze (10·5% Tin, 0·70% Phosphor).

Flux*	Phos. added %	Analyses of ingot: Tin %	Analyses of ingot: Phos. %	Ideal dens.	Obsd. bulk dens.	Porosity %	Appearance of ingot head	Properties of upper test piece: Dens. gm./cc.	Properties of upper test piece: U.T.S. ton/in.²	Properties of upper test piece: El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	Properties of lower test piece: U.T.S. ton/in.²	Properties of lower test piece: El% on 2 in.
NaCl 4%	0·1	10·5†	0·72	8·77	8·68	0·9	Trace exudation	8·68	24·0	17	8·72	24·0	15
3% SBO	0·1	10·5†	0·70	8·77	8·72	0·7	ditto	8·73	24·1	16	8·71	23·2	14
3% SB $\frac{1}{12}$	0·1	10·5†	0·68	8·77	8·74	0·3	clean	8·78	26·8	28	8·73	24·0	16
3%	0·1	10·5†	0·66	8·77	8·74	0·3	—	8·77	26·1	38	8·72	25·6	30
3% SB $\frac{1}{4}$	0·3	10·5†	0·70	8·77	8·75	0·2	—	8·80	26·0	22	8·77	25·0	19
3% $SB\frac{1}{3}$	0·3	10·2	0·78	8·76	8·80	—	—	8·81	28·9	42	8·79	27·6	29
3% $SB\frac{1}{2}$	0·3	10·1	0·82	8·75	8·75	—	—	8·80	26·6	24	8·73	26·5	25
None	—	10·8	0·75	8·76	8·68	1·0	Trace exudation	8·65	24·8	6	8·61	26·7	1

* Fraction denotes CuO content. Other constituents are sea-sand and fused borax in equal parts.
† Nominal.

further improvement in mechanical properties is obtained with flux SB$\frac{1}{2}$.

Oxidation Losses.—The analysis results show that with either gassy scrap or normal ingoted charges of 2B8 bronze, the phosphorus loss due to oxidation increases from 0·1% with the weakest fluxes to 0·4% with the richest flux tested. Tin losses increase similarly from 0·3% to 1%. Addition of the requisite amounts of tin and phosphor-copper, after removal of the oxidising flux, restores the bronze to its correct composition.

Degassing High Phosphorus Bronze (9% Tin, 1·5% Phosphorus)

Gassy Scrap Charges.—Charges of sand-cast scrap bronze containing 9% tin, 1·5% phosphorus, of high but varying initial gas content were treated with fluxes of increasing CuO content as for the 2B8 bronze. The results are summarised in Table VII and Fig. 4.

Due to the high initial gas content and the high phosphorus content complete degassing giving density and mechanical properties equal to virgin melts is achieved only with the rich fluxes containing $\frac{1}{2}$ CuO or more. Even the $\frac{1}{2}$ flux gave considerable variation in repeated results (See Fig. 4), and this was found to be related to variation in the gas content of charges. Very gassy charges could be completely degassed only by melting under the flux containing $\frac{1}{2}$ CuO.

Virgin Charges Intentionally Gassed.—In view of this variation it was decided to study the effect of CuO content of the flux on charges of constant initial high gas-content. Melts from virgin metals were prepared under the standard degassing flux (1 CuO, 1 sand, 1 borax) and after removal of this flux and addition of the phosphorus and tin, each melt was cooled to 1,200° C. and gassed by adding and stirring in 100 gms. of red foundry sand previously baked at 150° C. The bronze reacts with the combined moisture of the sand thus:

$$Me + H_2O \rightleftharpoons 2H + MeO$$

and the hydrogen goes into solution in the bronze.[7] To the gassed charge was added the flux to be tested. (In this case the fluxes had been pre-fused to remove any residual combined water from the borax, since this would interfere with the degassing action. With normal procedure such moisture is driven off before the bronze melts, and is thus harmless). The flux was allowed to react with the bronze for 10 minutes, then removed in the usual manner, and the calculated additions made for oxidation losses.

Tests were made normally with 6% flux additions, but some of the fluxes were also tested as 3% additions to determine the effect of total quantity, as distinct from the concentration of the CuO. The treated melts were poured slowly into the 2 in. diameter moulds, generally at 1,250° C., but some repeat tests were made with different pouring temperatures to determine the effect of this factor on the degree of degassing.

Blank tests on the method of gassing the virgin melts were made by pouring the gassed metal without further treatment into the 2 in. mould. The method gave consistently severely gassed ingots with 3%–3$\frac{1}{2}$% porosity, extensive internal cavities (Fig. 5) and white surface exudations.

Results of the tests on the treated ingots are given in Table VIII and Fig. 6. The sand-borax flux produces no appreciable degassing (2–3% porosity), but as the CuO content of the flux is increased, the porosity of the treated ingot is decreased, and mechanical properties are improved in a regular manner (Fig. 6). Complete degassing giving complete freedom from internal porosity (Fig. 7) and density and properties equal to those of degassed virgin melts are obtained in the ingots treated with fluxes containing 50% CuO or more. With the weaker fluxes there is considerable variation in the porosity and properties of the ingots according to the pouring temperature. (Table VIII). The points plotted in the graph are the mean values for the two tests with each flux.

The tests using only 3% instead of 6% flux, show that the total quantity of CuO available has little effect on the degassing compared with that of the concentration of CuO in the flux.

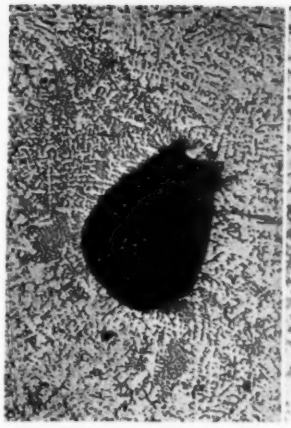


Fig. 5.—Gas hole and segregated compounds in gassed high phosphorus bronze (9% Sn, 1·5% P). × 100

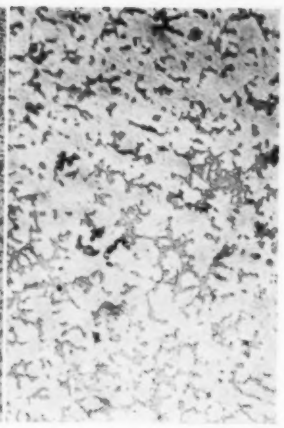


Fig. 7.—Degassed high phosphorus bronze (9% Sn, 1·5% P). ×150.

[7] Pell-Walpole, W. T., *Foundry Trades Journal*, Jan. 24, 31 to Feb. 7, 14, 21, 28, 1946.

TABLE VII.—Degassing Gassy Scrap Charges of High Phosphorus Bronze (1% Tin, 1·5% Phosphor.)

Flux* (3% of each)	Phos. added %	Analyses of treated ingot: Tin %	Analyses of treated ingot: Phos. %	Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Appearance of ingot head	Properties of upper test bar: Dens. gm./cc.	Properties of upper test bar: U.T.S. ton/in.²	Properties of upper test bar: El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	Properties of lower test piece: U.T.S. ton/in.²	Properties of lower test piece: El% on 2 in.
SBO	0·1	—	1·62	8·62	8·56	3·0	Severe exudation	8·39	19·9	3	8·30	18·0	3
SB$\frac{1}{16}$	0·3	—	1·43	8·65	8·48	2·0	Slight exudation	8·51	25·0	9	8·45	22·8	6
SB$\frac{1}{8}$	0·3	—	1·58	8·63	8·55	0·93	Trace exudation	8·57	27·6	19	8·51	25·1	12
SB$\frac{1}{4}$	0·1	—	1·35	8·66	8·65	0·11	Clean	8·64	28·3	29	8·62	24·2	11
SB$\frac{1}{4}$	0·0	—	1·18	8·69	8·68	0·1	—	8·69	24·8	10	8·64	22·4	5
SB$\frac{1}{4}$	0·3	—	1·53	8·63	8·59	0·5	—	8·61	29·2	24	8·59	27·9	21
SB$\frac{1}{4}$	0·3	—	1·46	8·65	8·56	1·0	—	8·56	27·6	20	8·52	22·8	8
SB$\frac{1}{4}$	0·3	—	1·53	8·63	8·62	0·1	—	8·64	29·0	24	8·61	24·4	12

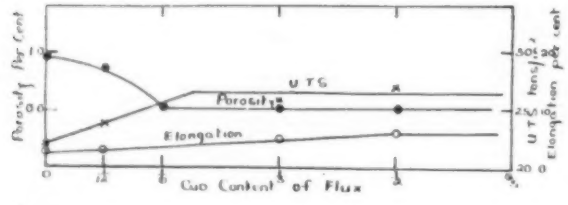
* Fraction denotes CuO content, other constituents are equal parts by weight of sea-sand and fused borax.

TABLE VIII.—The Effect of CuO Content of Flux, Amount of Flux and Pouring Temperature on the Properties of Ingots of High Phosphorus Bronze (9% Sn, 1·5% Phosphor) Prepared from Intentionally Gassed Virgin Charges.

Flux Charge*	Phos. added %	Pouring temp. °C.	Analyses of ingot: Tin %	Analyses of ingot: Phos. %	Ideal dens. gm./cc.	Obsd. bulk dens.	Porosity %	Appearance of ingot head	Properties of upper test piece: Dens. gm./cc.	Properties of upper test piece: U.T.S. ton/in.²	Properties of upper test piece: El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	Properties of lower test piece: U.T.S. ton/in.²	Properties of lower test piece: El% on 2 in.
6% SBO	—	1250	—	1·22	8·68	8·44	2·8	Severe exudation	8·49	24·0	10	8·38	19·6	4
6% SB$\frac{1}{10}$	0·1	1200	—	1·47	8·64	8·53	1·3	Some exudation	8·53	25·8	14	8·46	23·1	9
6% SB	0·1	1300	—	1·50	8·64	8·46	2·0	Severe exudation	—	—	—	8·43	20·4	5
6% SB$\frac{1}{2}$	0·3	1150		1 50	8·64	8 62	0·2	Clean	8·63	28·0	24	8 57	27·4	11
6% SB$\frac{1}{2}$	0·1	1250	—	1·39	8·66	8·51	1·7	Slight exudation	8·55	24·9	12	8·47	24·6	11
6% SB$\frac{1}{2}$	0·3	1300	—	1·38	8·66	8·56	1·1	Trace exudation	8·61	27·6	22	8·48	24·4	16
6% SB$\frac{1}{4}$	0·3	1200	—	1·40	8·66	8·60	0·7	Clean	8·62	25·9	14	8·55	24·9	11
6% SB$\frac{1}{4}$	0·3	1250	—	1·26	8·68	8·72	—	Clean	8·76	28·7	44	8·70	26·0	21
3% SB$\frac{1}{2}$	0·3	1250	9·3	1·46	8·65	8·54	1·3	Trace exudation	8·56	27·3	18	8·50	24·2	10
3% SB$\frac{1}{2}$	0·3	1250	9·2	1·48	8·64	8·61	0·3	Clean	—	—	—	8·62	26·0	14
3% SB$\frac{1}{2}$	0·5	1250	—	1·72	8·60	8·58	0·2	Clean	8·56	28·8	26	8·57	26·9	15
3% SB$\frac{1}{4}$	0·5	1250	—	1·70	8·61	0·0	—	—	8·639	27·4	21	8·59	27·0	20
3% SB$\frac{1}{4}$	0·3	1250		1·50	8·64	8·60	0·5	Clean	8·62	25·9	14	8·55	24·4	11
3% SB$\frac{1}{4}$	0·3	1250	—	1·50	8·64	8·63	0·1	Clean	—	—	—	—	—	—
3% SB$\frac{1}{4}$	0·3	1250	—	1·39	8·66	8·65	0·1	Clean	8·66	27·6	18	8·61	24·0	12

* Fraction indicates CuO content of flux. Other constituents equal parts of sea-sand and fused borax.

Fig. 8.—The effect of CuO content of flux on ingots of high phosphorus (11% Sn, 1.5% P) bronze prepared from ingoted charges of low initial gas content.



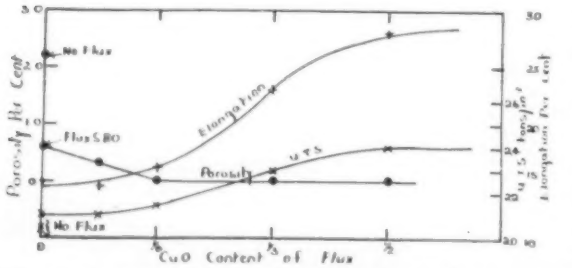


Fig. 9.—The effect of CuO content of the flux on the porosity and properties of ingots of medium phosphorus bronze (9% Sn, 0.4% P) prepared from gassy sand-casting charges.

Ingoted Charges of Low Initial Gas Content.—Ingoted charges of bronze containing the same phosphorus content (1·5) but rather higher tin contents (11% nominal) prepared from virgin metals and having low gas content, were treated as before with fluxes of various CuO contents, and cast into 2 in. ingots as before.

Results of tests on the treated ingots (Table IX and Fig. 8) show that due to the initial low gas content of the charges, complete freedom from gas is obtained by melting under the weak flux containing $\frac{1}{6}$ CuO. Tensile properties of this series are lower than the preceding ones (except for the 9% tin ingot produced with flux SB $\frac{1}{2}$) due to higher tin contents. A degassed virgin melt containing 11% tin with 1·5% phosphorus gave 24·3 tons/in.², 3½% elongation.

The phosphorus losses for the various fluxes are similar to those given previously. The effect of variation in the phosphorus contents on the density of the ingots was taken into account in determining the percentage porosity. Tin content has no appreciable effect on the "ideal" density.

Degassing Medium-Phosphorus Bronze (8–9% Tin, 0·3–0·4% Phosphorus)

A similar series of tests was made with charges of scrap from gassy sand-cast phosphor-bronze billets containing 8–9% tin, 0·3–0·4% phosphorus. The results (Table X, Fig. 9) show that due to the lower phosphorus content porosity is eliminated by melting under the weak flux containing $\frac{1}{6}$ CuO, although some slight further improvement in mechanical properties is obtained by melting under the stronger flux containing $\frac{1}{4}$ CuO. It is probable that in the ingot treated with flux CuO $\frac{1}{4}$ there is slight residual porosity of the fissure type. This would have little effect on density but would reduce mechanical properties appreciably. By melting under the next richer flux ($\frac{1}{3}$ CuO), this last trace of gas is eliminated with consequent restoration of mechanical properties to the values obtained with degassed virgin melts.

TABLE IX.—The Effect of CuO Content of the Flux on the Porosity and Properties of Ingots of High Phosphorus Bronze (11% Tin, 1·5% Phos.) Prepared from Ingoted Charges of Low Initial Gas Content.

Flux Charged*	Phos. added %	Analyses of treated ingots: Sn %†	Analyses of treated ingots: Phos. %	Ideal dens. gm./cc.	Obsd. bulk dens.	Porosity %	Properties of upper test piece: Dens. gm./cc.	Properties of upper test piece: U.T.S ton/in.²	Properties of upper test piece: El% on 2 in.	Properties of lower test piece: Dens. gm./cc.	Properties of lower test piece: U.T.S. ton/in.²	Properties of lower test piece: El% on 2 in.	Appearance of ingot head
SBO	0·1	11·0	1·55	8·63	8·53	1·2	8·55	22·0	4	8·51	22·0	2	Moderate exudation
SB$\frac{1}{10}$	0·1	11·0	1·55	8·63	8·57	0·7	8·57	25·6	4	8·52	22·0	2	Slight exudation
SB$\frac{1}{2}$	0·3	9·0	1·64	8·62	8·64	—	8·67	28·6‡	26	8·63	27·0‡	18	Clean
SB$\frac{1}{2}$	0·3	11·0	1·60	8·62	8·65	—	8·64	24·8	5	8·61	24·8	2	Clean
SB$\frac{1}{4}$	0·3	11·0	1·43	8·65	8·68	—	8·62	28·5	8	8·60	23·7	3	Clean
SB$\frac{1}{4}$ with virgin charge	—	11·0	1·50	8·64	8·67	—	8·65	25·7	4	8·65	22·8	3	Clean

* Fraction denotes CuO content. Other constituents are equal parts of sea-sand and fused borax.
† Nominal.
‡ High tensile values due to lower tin content.

TABLE X.—THE EFFECT OF CUO CONTENT OF THE FLUX ON THE PROPERTIES OF INGOTS OF MEDIUM-PHOSPHORUS BRONZE (9% Sn, 0·35% PHOS.). PREPARED FROM SAND-CAST SCRAP CHARGES.

Flux Charge* (3% of each)	Phos. added %	Analysis of treated ingots		Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Tin %	Phos. %				Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
SBO	0·1	9·8	0·34	8·82	8·78	0·6	8·78	22·3	18	8·78	19·7	11
SB$_{\frac{1}{2}}$	0·2	—	0·37	8·83	0·81	0·3	8·82	22·4	16	8·80	19·6	11
SB$_{\frac{1}{3}}$	0·2	—	0·32	8·83	8·83	0	8·84	23·1	22	8·80	19·6	8
SB$_{\frac{1}{6}}$	0·2	9·5	0·39	8·82	8·85	0	8·85	24·3	28	8·84	22·7	18
SB$_{\frac{1}{12}}$	0·2	—	0·37	8·82	8·84	0	8·80	25·5	30	8·80	22·5	25
None	0·1	—	0·32	8·85	8·62	2·2	8·60	20·0	10	8·66	21·0	12

* Fraction denotes CuO content. Other constituents equal parts by weight of sea-sand and fused borax.

One charge of this material melted without cover gave an ingot with higher porosity and poorer mechanical properties than any of the flux-treated ingots (Table X).

Control of Gas Content in Admiralty Gun Metal

Preparation of Charges of High Gas Control.—Work has been reported previously[6] which shows that ingoted scrap Admiralty gun metal can be efficiently degassed by melting under the flux containing $\frac{1}{6}$ CuO (See figures quoted in Table XI). It remained to determine the conditions required to degas charges of high initial gas content. Attempts were made to produce gassy metal by adding red sand to the molten virgin metal charges, as was done with high phosphorus bronze. This method gave inconsistent results with the gun metal, however, probably due to the interfering effect of the surface oxide skin. Charges of controlled high gas content were then prepared by pouring the molten degassed alloy through a Salamander dozzle lined with red sand and baked at 100° C. This gave a series of ingots having very low densities (8·40–8·60) and showing severe internal gas porosity (Fig. 10) and yellowish-white exudations. The gassy ingots were cut into two and the density of each half was determined. Then by suitable mixing of halves from different ingots it was possible to make up charges of gassy metal of density 8·52 ± 0·03 gm/cc., i.e., with $\sim$ 3% gas porosity.

Degassing Ingoted Charges of High Gas Content.—Charges of the gassed gun-metal were melted under the sand-borax flux, and under 3% or 6% charges of oxidising fluxes containing $\frac{1}{12}$, $\frac{1}{6}$, $\frac{1}{3}$ or $\frac{1}{2}$ CuO by weight, and in air (oxidising flame) without flux cover. With all oxidising flux treatments an addition of $\frac{1}{2}$% zinc and $\frac{1}{2}$% tin was made after removal of the flux to compensate for oxidation losses. The melts were poured at 1,200° C. through a 9/64 in. aperture in a clean pre-heated Salamander pot (pouring rate = 10 lb./minute) into the thin-walled 2 in. diameter chill mould. These conditions are known to give freedom from porosity in degassed virgin melts.[6]

The ingots were examined and tested as described for phosphor-bronze billets. The results are summarised in Table XI and Fig. 11. The sand-borax flux gave an appreciable decrease in porosity (to 1·6%) probably due to escape of gas from the metal and through the flux during melting. The ingot from this treatment showed no visible exudation but microscopical examination showed extensive fissure porosity and some cavities. The tensile strength was only 17 tons/in.²

Fig. 11.—Degassing gassed charges of 88–10–2 gun metal.

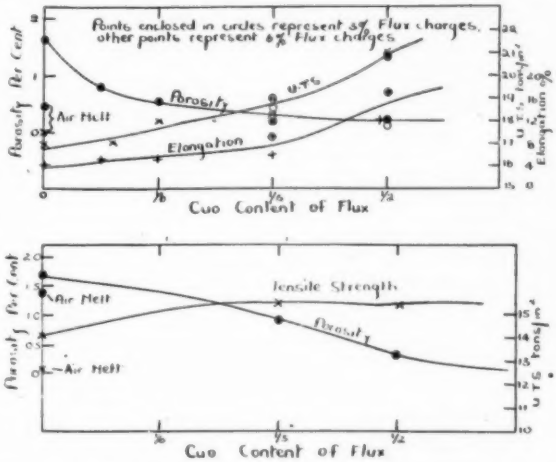


Fig. 14.—The effect of melting conditions on porosity and tensile strength of ingots of high lead bronze (20% Pb, 5% Sn.) prepared from gassed charges.

Melting under fluxes of increasing CuO content produced a rapid increase in density but only a slow improvement in mechanical properties until the flux contained $\frac{1}{3}$ CuO by weight (Fig. 11). The porosity with this flux is very low, but the tensile properties (19 tons/in.², 12% elongation) are poor compared with those obtained with degassed virgin melts (21–22 tons/in.,² 20–25% elongation). Further increase of the CuO content of the flux produced little further increase in density but tensile properties increased to maximum

TABLE XI.—DEGASSING SCRAP CHARGES OF 88/10/2 GUN METAL.

Flux Used*	Porosity of charge %	Additions		Analysis of treated ingot			Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Sn %	Zn %	Sn %	Zn %	Pb%				Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
6% SBO	3·4	0·5	0	10·5	2·3	—	8·83	8·69	1·6	8·71	17·8	5	8·65	16·2	3
6% SB$_{\frac{1}{2}}$	3·6	0·5	0·5	10·2	2·7	—	8·82	8·75	0·8	8·80	17·8	6	8·74	16·2	3
6% SB$_{\frac{1}{3}}$	3·2	0·5	0·5	10·6	2·8	—	8·82	8·77	0·55	8·76	17·7	4	8·81	18·4	5
6% SB$_{\frac{1}{6}}$	3·5	0·5	0·5	10·6	2·4	—	8·83	8·81	0·2	8·79	18·4	7	8·74	17·9	5
6% SB$_{\frac{1}{12}}$	3·7	0·5	0·5	10·5	2·2	—	8·83	8·81	0·2	8·81	22·9	17	8·78	19·2	8
3% SB$_{\frac{1}{3}}$	3·5	0·5	0·5	10·5	2·3	—	8·83	8·79	0·45	8·83	19·6	10	8·76	18·5	8
3% SB$_{\frac{1}{6}}$	3·5	0·5	0·5	10·3	2·2	—	8·83	8·82	0·1	8·78	21·8	20	8·78	20·8	15
None. Oxidising flame	3·4	0·5	0·5	11·1	2·4	—	8·83	8·80	0·34	8·80	19·1	8	8·80	15·9	1
None. Oxidising flame	3·4	0·5	0·5	10·5	2·2	—	8·83	8·78	0·6	8·81	17·8	6	8·81	17·6	5

* Fraction denotes CuO content of flux. Other constituents are equal parts of sea-sand and fused borax.

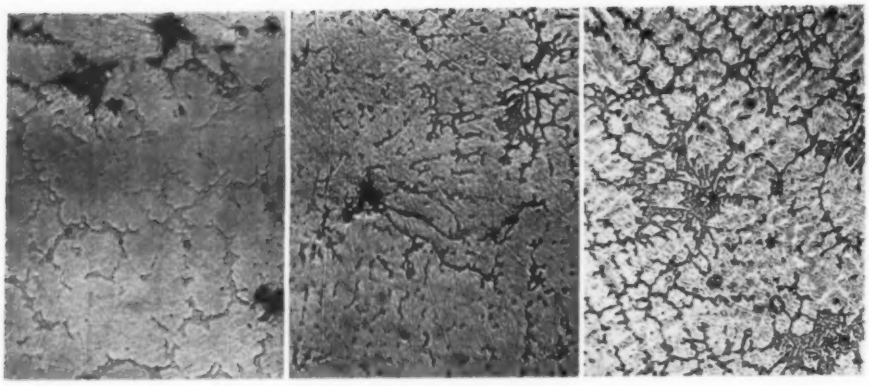


Fig. 10.—Gas porosity in intentionally gassed gun metal. × 150

Fig. 12.—Fissure porosity in partially degassed Admiralty gun metal, 88–10–2, melted in air and poured slowly. × 150

Fig. 13.—Degassed Admiralty gun metal, 88–10–2, melted under slowly oxidising flux, free from fissures. × 150

values (Fig. 11). This behaviour is similar to that noted with bronzes of low or medium phosphorus content. The microstructures show that with fluxes containing up to $\frac{1}{3}$ CuO major cavities are eliminated but considerable fissure porosity remains (as Fig. 12). These fissures have little effect on density but seriously impair mechanical properties. Complete degassing, obtained by melting under the flux containing $\frac{1}{2}$ CuO eliminates these fissures (Fig. 13) and restores the mechanical properties to the maximum values obtainable with degassed virgin melts.

Variation of the quantity of oxidising flux had more effect on the efficiency of degassing gun metal than was found with phosphor bronze, but was still less important than the concentration of CuO in the flux. This effect is to be expected, since the degassing reactions can occur only at the flux-metal interface and the reactions are allowed to proceed for only a limited time. The total quantity of flux present is only likely to be important in cases where the amount of gas to be removed (or the amount of zinc, etc., oxidised) is so great that the CuO content of a small flux charge might become exhausted before degassing was completed.

Melting without flux, but with an oxidising flame, similarly removes most of the gas from the gassed charge, e.g., density is increased from 8·52 to 8·78 and all coarse porosity is eliminated from the microstructure, but extensive fissuring remains, in this case associated with entrapped oxide films (Fig. 12). The mechanical properties are consequently very inferior to those obtained with the rich oxidising flux.

Zinc Loss.—Zinc loss was negligible with air melting or with fluxes containing up to $\frac{1}{6}$ CuO by weight. Melting under more strongly oxidising fluxes caused a loss of 0·2–0·4% zinc. The addition of $\frac{1}{2}$% zinc made after removal of the flux was therefore excessive with fluxes of low CuO content, but was satisfactory with the rich flux containing $\frac{1}{3}$ CuO which is necessary to ensure complete degassing.

Control of Gas Content in High-Zinc Gun Metal

Gassy charges of high zinc gun metal (7% tin, 5% zinc) were prepared by the same method as for Admiralty gun metal, but the degree of gassing obtained was slightly less (density 8·55, porosity 2·5%). This is probably due to the effect of the high zinc content ensuring a higher zinc vapour pressure in the molten metal, thus reducing the solubility for hydrogen. The gassy ingots were remelted either in air, under neutral sand-borax flux, or under oxidising fluxes of various CuO contents, and were cast into 2 in. diameter ingots under the same conditions as the Admiralty gun metal. The properties of the ingots are summarised in Table XII.

This alloy is degassed much more readily than the low-zinc gun metal. Melting either in air or under the neutral flux gave fairly complete degassing, with densities equal to those obtained with the oxidising flux. Complete degassing and maximum mechanical properties are obtained only by melting under very strongly oxidising flux ($\frac{2}{3}$ CuO). The zinc loss is appreciably greater than with 88/10/2. It increases from 0·3% for the sand-borax flux to 1·3% with the rich oxidising fluxes. The losses on melting without cover were high and rather erratic. There is no appreciable tin loss, the tin being " protected " from oxidation by the presence of the zinc.

Control of Gas Content in Leaded Gun Metal

Gassy charges of leaded gun metal (85/5/5/5) were prepared by the method used for the lead-free gun metals, but, in agreement with previous experience of this type of alloy[7], less gas was absorbed from the reaction with the sand. The gassed ingots had an average density

TABLE XII.—Degassing Gassed Charges of High Zinc Gun Metal. (88/7/5).

Flux Used*	Porosity of charge %	Additions		Analysis of ingot treated			Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Sn %	Zn %	Sn %	Zn %	Pb %				Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
3% SBO	2·6	0·5	0	7·4	4·9	—	8·78	8·78	0	8·83	21·8	15	8·78	19·9	22
6% SBO	2·6	0·5	0	7·7	5·0	—	8·78	8·76	0·2	8·76	22·0	28	8·83	13·0	12
3% SB⅓	2·6	0·5	0·5	7·5	5·3	—	8·78	8·77	0·1	8·78	23·0	44	8·79	19·9	22
3% SB½	2·6	0·5	0·5	7·8	4·6	—	8·79	8·79	—	8·80	21·2	29	8·78	—	—
3% SB⅔	2·6	0·5	0·5	7·6	4·6	—	8·79	8·78	0·1	8·77	21·0	22	8·75	18·8	17
5% SB⅓	3·7	0·5	0·5	7·4	4·7	—	8·78	8·83	0	8·80	22·8	41	8·80	20·2	21
None. Oxidising flame..	2·6	0·5	0	7·7	4·9	—	8·78	8·79	0	8·76	20·9	22	8·73	17·1	30
None. Oxidising flame..	3·7	0·5	0	7·6	1·2	—	8·80	8·78	0·2	—	—	—	8·80	22·1	22

* Fraction denotes CuO content of flux. Other constituents are equal parts by weight of sea-sand and fused borax.

TABLE XIII.—Degassing Gassed Charges of 85/5/5/5 Leaded Gun Metal.

Flux Used*	Porosity of charge %	Additions		Analysis of treated ingots			Ideal dens. gm./cc.	Obsd. bulk dens. gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Sn %	Zn %	Sn %	Zn %	Pb %				Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.	Dens. gm./cc.	U.T.S. ton/in.²	El% on 2 in.
3% SBO	2·6	0·5	0	5·5	4·5	4·5	8·87	8·82	0·56	8·86	19·6	37	8·77	16·1	18
3% SB½	2·6	0·5	0·5	5·7	5·2	5·2	8·86	8·86	0	8·85	17·8	19	8·84	16·6	19
3% SB¼	2·6	0·5	0·5	5·6	5·2	5·0	8·86	8·85	0·13	8·84	18·2	19	8·85	17·8	17
None. Oxidising flame..	2·6	0·5	0	5·6	3·8	5·4	8·88	8·85	0·35	8·90	19·0	19	8·82	17·6	22

* Fraction denotes CuO content of flux. Other constituents are equal parts by weight of sea-sand and fused borax.

of 8·63 gm/cc. compared with 8·86 for degassed virgin melts (≃ 2·47 porosity). The gassed ingots were remelted either in air (oxidising flame) or under neutral or oxidising fluxes of the sand/borax type, and poured into 2 in. ingots under the same conditions as those used for the lead-free gun metals, tested as before (Table XIII).

The gassed metal lost most, but not all, of its gas by remelting under the sand-borax flux. This treatment gave an ingot with density 8·82. The weak oxidising flux gave high density but poor properties. Treating with a strongly oxidising flux (CuO ½) or melting without cover, but with an oxidising flame, gave complete degassing (density 8·85–8·86 gm/cc.). The mechanical properties were slightly inferior to those of degassed virgin melts. No loss of either lead or tin content occurred with any of the treatments, but a high zinc loss resulted from melting without cover, in an oxidising flame, and a slight zinc loss occurred with oxidising fluxes.

Control of Gas Content in Zinc-free Leaded Bronzes

10% *Tin*, 10% *Lead*, 0·1 *Phosphorus*.—Gassy charges of leaded bronze of this composition were prepared as for gun metals, by pouring the degassed molten alloy through a Salamander dozzle lined with red sand. The extent of gas absorption was much less than with the lead-free gun metals, in agreement with previous experience of alloys of high lead content. The gassed ingots, of density 8·77 gms/cc. (approximately 2% porosity), were remelted under neutral oxidising sand-borax fluxes, or without cover in an oxidising flame, and poured into 2 in. moulds as before. The properties of the ingots are summarised in Table XIV.

Partial degassing was obtained by melting under the sand-borax flux or under mildly oxidising fluxes and complete degassing (density 8·97) by melting either without cover but in an oxidising flame, or under a strongly oxidising flux (containing ½ CuO). No tin loss occurred in any treatment, but approximately 1% lead was lost with each method.

20% *Lead*, 5% *Tin*, 0·1% *Phosphorus*.—Gassy charges of this composition obtained by pouring the molten metal through a sand-lined funnel) had less than 2% porosity and alternative methods of producing gassy metal were tried as follows: (*a*) addition of red sand to the molten metal as used for high-phosphorus bronzes, (*b*) melting under a reducing flame. Charges gassed by one of these methods were then remelted either in air, or under sand-borax flux or under oxidising fluxes. Ingots were cast as before and were examined for porosity and tensile properties (Table XV, Fig. 14).

The ingot from the melt treated with the sand-borax flux had appreciable porosity and poor mechanical properties. The flux containing ⅓ CuO gave fairly complete degassing and high mechanical properties, but complete freedom from porosity was obtained only with the rich oxidising flux, SB½. Melting in air with an oxidising flame gave only partial degassing and very poor mechanical properties.

Summary and Conclusions

Bronzes of relatively high phosphorus, zinc or lead contents can be degassed by melting under fluid oxidising fluxes. The degassing probably occurs by oxidation reaction at the flux-metal interface. Slight oxidation losses of phosphorus, zinc, or lead are incurred at the same time, but these are generally lower and more easily controlled than losses which occur by melting without flux.

Ingoted charges of phosphorus bronzes containing

TABLE XIV.—Degassing Gassed Charges of Leaded Bronze. (10% Lead, 10% Tin).

Flux Used*	Porosity of charge	Analysis of treated ingot		Ideal Density gm./cc.	Observed bulk density gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Lead %	Tin %				Density gm./cc.	U.T.S. ton/in.²	IE % on 2 in.	Density gm./cc.	U.T.S. ton/in.²	El % on 2 in.
3% SBO	2·6	9·0	10·5	9·01	8·93	0·9	8·92	17·1	6	8·89	16·1	7
3% SB½	2·6	9·0	10·4	9·01	8·93	0·9	8·92	16·5	7	8·86	15·5	6
3% SB¼	3·6	9·0	10·7	9·01	8·97	0·4	8·98	17·1	9	8·97	16·8	6
None. Oxidising flame..	2·6	9·2	10·4	9·01	8·97	0·45	8·92	18·1	11	8·97	15·9	8

* Fraction denotes CuO content. Other constituents are equal parts by weight of sea-sand and fused borax.

TABLE XV.—Degassing Gassed Charges of High-lead Bronze. (20% Lead, 5% Tin).

Flux Used*	Porosity of charge	Analysis of treated ingot		Ideal density gm./cc.	Observed bulk density gm./cc.	Porosity %	Properties of upper test piece			Properties of lower test piece		
		Lead %	Tin %				Density gm./cc.	U.T.S. ton/in.²	El % on 2 in.	Density gm./cc.	U.T.S. ton/in.²	El % on 2 in.
6% SBO	†	19·8	5·0	9·20	9·04	1·7	9·05	14·9	18	8·99	13·8	19
3% SB½	†	18·5	5·1	9·18	9·10	0·9	9·11	15·9	14	9·06	15·4	16
3% SB¼	2·2	18·0	5·2	9·16	9·13	0·3	9·11	15·3	14	9·10	15·2	15
None. Oxidising flame..	1·5	22·8	5·2	9·25	9·12	1·4	9·18	12·5	16	9·12	12·8	17

* Fraction denotes CuO content. Other constituents are equal parts by weight of sea-sand and fused borax.
† Charge gassed by melting under reducing flame then adding red-sand prior to treatment with flux.

0·3–1·5% phos. of relatively low initial gas content (i.e., free from exudations or visible signs of porosity) require a flux containing CuO 1, fused borax 1, sharp sand 1. Very gassy charges of these metals require a richer flux containing CuO 2, fused borax 1, sharp sand 1. The melting losses due to oxidation are 0·3% phosphorus, 0·5% tin for the weaker flux, 0·4–0·5% phosphorus, 0·8–1·0% tin for the stronger flux.

Ingoted charges of Admiralty and other gun metal of low initial gas content can be successfully degassed by melting under a flux containing CuO½, fused borax 1, sand 1 parts by weight. The zinc loss on oxidation varies from 0·2%–0·5% according to initial zinc content. Scrap charges of Admiralty gun metal of high initial gas content require a flux containing CuO 2, fused borax 1, sharp sand 1. Melting in an oxidising flame without cover gives only partial degassing and inferior properties. The zinc loss is 0·5–0·7% with the flux CuO 2, sand 1, fused borax 1.

Gassy charges of high-zinc gun metals are degassed most efficiently by melting under a very strongly oxidising flux (CuO 4, sand 1, fused borax 1), but fairly complete degassing is obtained by melting in an oxidising atmosphere without flux or under a neutral weak oxidising flux (sand 5, borax 5, CuO 1). The zinc loss is 1–1·3, with air melting or with the rich flux, only 0·3–0·5% with the neutral or weakly oxidising fluxes.

Gassy leaded gun metal charges can be degassed equally well by melting in an oxidising flame without cover, or under a strongly oxidising flux (CuO 2, sand 1, fused borax 1), but the flux process ensures a much lower zinc loss (0·5%) than the melting in air (1½–2%). No appreciable loss of lead or tin occurs.

Ingoted charges of leaded bronzes can be degassed either by melting with an oxidising flame or with a weak oxidising flux. Gassy scrap charges of leaded bronze containing 10% tin, 10% lead require a similar treatment to gassy leaded gun-metal, but with high lead bronze (20% lead, 5% tin) melting in air gives unsatisfactory results, and a rich oxidising flux (CuO 2, sand 1, borax 1) alone gives complete degassing. There is no appreciable tin loss with these alloys, but lead loss is from 1–2% (according to lead content) when strongly oxidising fluxes are used.

The treatments recommended above for "complete degassing" of the various alloys give ingots which are free from harmful types of porosity, and which have mechanical properties equal to those obtained from degassed virgin melts. In all cases of treatment by use of oxidising fluxes the amount of flux used should be sufficient to give a fluid seal (2–3% of the charge) and the crucible should be thoroughly impregnated with the flux before use.

The recommended additions for oxidation losses must be added *after* removal of the degassing flux. The actual amounts required may vary slightly according to furnace conditions, temperature, etc., but the figures quoted should serve as a general guide to the relative order of losses to be expected.

Acknowledgments

This work was carried out in collaboration with the Tin Research Institute and the author's thanks are due to Dr. K. Winterton, formerly of the Institute's staff, for help with part of the experimental work and to Dr. W. J. Price of the Institute's staff for the chemical analyses.

Nickel and its Alloys in 1947

THE review of the industry by Mr. Robert C. Stanley, Chairman and President of The International Nickel Company of Canada Limited, bears out the promise given in his survey at the end of 1946. Nickel sales are still expanding, world deliveries of Canadian nickel in all forms for the year 1947 will be in excess of those of 1946. Canadian nickel deliveries in 1947, in all markets, will approximate those of the industry's greatest peace-time year of 1937, and shipments in the United States and Canada will show an increase of about 50% over 1937.

Operations of International Nickel at Copper Cliff, its mining and smelting centre, were substantially higher than in 1946, despite the continuing labour shortage. Nickel prices in the U.S. and U.K. remained unchanged during the year. On November 21, the International Nickel Company, Inc., announced a reduction of 1¼ c. per pound in its price for refined nickel in the United States. The reduction becomes effective January 1, 1948, simultaneously with a like reduction in the United States import duty on refined nickel. The prices of nickel outside the United States are not affected by the duty and remain unchanged.

Mr. Stanley confines his review to nickel and its alloys, indicating many new trends of consumption. Of great interest is the growing utilisation of high strength low alloy steels which contribute materially to the over-all consumption of nickel by the steel industry. Nickel alloy steels of the pre-war standard types have re-established salient positions in industry both in the U.K. and on the Continent, where they are once more taking a prominent part in the new steel specifications.

The past year saw a further broadening of the uses of Monel, Inconel and other nickel alloys, as well as rolled nickel. The Nimonic alloys continue to be the standard turbine material for all British jet-propulsion engines, and the expanding field of electronics has brought many new uses of nickel and nickel alloys, such as in television.

Throughout the world the general trend towards further use of pure nickel and cupro-nickel for coinage purposes has accounted for increased tonnage. The demands for most of the established applications of nickel-electro-deposits have continued to increase, and the amount of nickel used for plating purposes is still maintaining a steady high level.

The encouraging outlook for business in the many industries which nickel serves indicates a continuance of the favourable showing made by the nickel industry in 1947.

B.O.C. Welding Prize

THE Council of the British Welding Research Association have gratefully accepted an offer from Messrs. British Oxygen Company Ltd., to provide a prize fund of £100 per annum for three years for a competition relating to welding.

A single prize of £100 is offered in 1948 and will be awarded for the best paper submitted on a research into welding or its application. Papers will require to be submitted on or before September 30, 1948.

Hydrogen, Nitrogen and Oxygen in Ferrous Metals

Their Properties and their Determination—Part III

By E. C. Pigott

*A very extensive literature exists on the gaseous constituents of irons, steels and ferro-alloys. Over a period of many years leading chemists and metallurgists have been engaged in piecing together the intricate facts brought to light. It would now seem that the main experimental and co-ordinating work has been done and that a clear perspective of this once totally obscure field has become possible. Such a perspective was commenced in our October, 1947, issue and the present article reviews the procedure for determining nitrogen contents.**

Consideration of Methods

It is in its combination as nitrides that nitrogen calls for primary consideration in ferrous analysis since, except for small proportions of cyanogen compounds and negligibly minute amounts of the element in the gaseous condition, all the nitrogen present in iron and steel exists in the form of nitrides of the various constituents, usually with iron nitride and manganese nitride predominating.

Three fundamentally different procedures are available:—

1. Total extraction in the gaseous state;
2. conversion into an ammonium salt, and alkali distillation;
3. conversion into an ammonium salt, and direct colorimetric finish.

The first of these, which yields both combined and uncombined nitrogen, most frequently takes the form of melting the sample in vacuo in a graphite crucible and analysing the extracted gases, a process that is satisfactorily accurate even in the presence of the most stable nitrides – namely, those of chromium, titanium, vanadium, tungsten, tantalum and niobium. Normally, the bulk of the nitrogen is found in the final, alumina, fraction; the method is not subject to interference from manganese vapour. Certain steels require a particularly high vacuum as well as prolonged evacuation after the oxygen-bearing gases have been removed. When these requirements are not met, the method tends to yield slightly low results.

Vacuum fusion analyses exposed the possibility of errors in other methods when these were undergoing their teething troubles. Unfortunately the vacuum-fusion apparatus is costly and calls for an expert operator, while a determination may last several hours. Various modifications of the process have been developed in this country, in the United States and in Germany. Klinger assists decomposition of the nitrides by the use of a tin-antimony flux. The degree of accuracy for these several modifications is of the order of $\pm 0{\cdot}0015\%$ of nitrogen.

The second means of approach – that of conversion into an ammonium salt by decomposition of the sample in mineral acid and of distillation of the ammonia from alkaline solution—is the one most generally used. It had its inception in 1880 at the hands of A. H. Allen (J.I.S.I., **8**, 181), who developed a procedure practised by Boussingault (Comptes Rendus, 1861, **53**, 77) and utilised Nessler's reaction.

A voluminous literature has grown from the necessity of ensuring complete decomposition of the nitrogen compounds. Where all the nitrides present are readily soluble, the procedure is straightforward and in the case of carbon steels, for example, usually yields results comparable with those obtained from the best vacuum-fusion technique. Indeed, the chemical distillation method has been acclaimed as being the more accurate. Many of the erroneous values obtained in the past were due to the insolubility of the nitrides of vanadium, titanium, silicon, aluminium, etc., in hydrochloric acid, and others, to excessively high blanks.

For many years it was the invariable practice to treat the insoluble residue separately. Boussingault's method was to fuse it in soda lime; this was early abandoned in favour of digestion with conc. sulphuric acid and potassium sulphate, sometimes in the presence of a catalyst. Johnson recommended sulphuric acid and perchloric acid for the purpose, a mixture that has been applied also as the initial solvent, in some cases with selenium, as a means of obviating a separate treatment. To this same end, initial solvents of sulphuric and perchloric acids, or sulphuric acid and potassium bisulphate are much used in the United States, where, for stainless materials, Cunningham resorts to a small addition of hydrofluoric acid to the far more conventional initial solvent of hydrochloric acid. Dissolution of high-silicon materials is much promoted by the addition of a little potassium fluoride. The length of the minimum digestion-time increases with rising carbon content and is greatest in the cases of 4% silicon transformer steel and high-titanium stainless steel.

Most commercial steels respond completely to the simple initial solvent of hydrochloric acid. In the presence of titanium, which modifies the composition of the nitrogen compounds, the normal hydrochloric acid treatment, no matter how applied, yields low results and requires modification. It appears best to use sulphuric acid and to fume for fifteen minutes or so, a simple expedient that is entirely effective in decomposing not only the titanium compounds, but also various other compounds not attacked by hydrochloric acid. Nevertheless, with highly complex alloys, it is still advisable to treat any residue separately.

*Continued from November issue, page 27.

Of considerable importance is the physical condition of the sample, especially in the case of alloy irons and steels, in which the mode of occurrence of the element can be converted, by annealing, into compounds that are insoluble in hydrochloric acid and sulphuric acid alike. The annealing of a 4·5% Ni—1·25% Cr steel at 650° C. followed by slow cooling, reduced the chemical yield from 0·013% nitrogen to a mere 0·005%. Due regard must be paid to this very influential factor, especially where the history of the material is unknown.

Provided decomposition of the sample is effected by not more than moderate heating, no loss of ammonia ensues.

The fact that any free nitrogen, dissolved or absorbed, or mechanically entrapped in blowholes, fails to be recorded by the distillation method, has been grossly over-emphasised, since the proportions in question are so immeasurably small as to fall well within the limits of the best experimental error. It has been claimed that most of such nitrogen may be brought into a recordable state by means of heat-treatment, a theory that cannot yet be accepted.

An essential requirement in operating the method is an ammonia-free atmosphere; usually, therefore, the main laboratory is unsuitable, and it is advisable to use a small special room carefully protected from ammoniacal contamination. A sometimes unsuspected source of extraneous ammonia is the wearing of laboratory coats that have been worn for some time in the main laboratory.

As a preliminary, it is necessary to wash the sample with water and with organic solvents in order to remove possible ammonia and grease films.

All solutions must be made up from nitrogen-free water; they are preferably kept in bottles having fume caps. Frequent blank determination and standard steel determinations are required.

Steam-distillation, which permits the use of larger samples without excessive bumping, holds promise as a means of speeding up the method.

The ammonia collected by distillation is determined either by titration with very dilute acid, or colorimetrically using the Nessler reaction. The former is entirely satisfactory, except for very low nitrogen contents. A mixed indicator of methyl-red and methylene blue provides a sharper end-point than methyl-red alone; an indicator preferred by some authorities is sodium alizarin sulphonate, while still others favour potentiometric titration, using a *p*H meter with glass electrode.

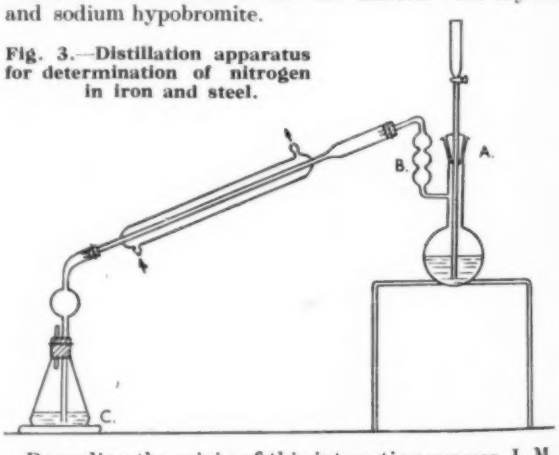
The Nessler finish usually consists in visually comparing the colour intensity of the test with that of a solution containing a known amount of a standard ammonium chloride solution. Rapid colour comparison is facilitated by the use of a colorimeter or, better still of a Fisher, Helige or B.D.H. nesslerimeter. If a photoelectric absorptiometer is employed, all that is necesary is to read off the content from a calibration graph. Certain errors are inherent in the Nessler method. The tint obtained is affected by the carbon present in the sample; the explanation would appear to be that the presence of carbon gives rise to the formation of amines. When the carbon content exceeds about 0·25% the interference can cause erroneous results. Variations in tint accrue in the comparison solution if the Nessler reagent is added before instead of after the standard solution of ammonium chloride. The most satisfactory procedure is to have the standard contain approximately the same amount of ammonia as the test and to equalise by diluting the more intense solution. Nesslerising is not applicable where the nitrogen content is high. The distillate should contain, per 100 ml., not more than 0·06 mg. of ammonia and about 2 ml. of Nessler reagent. About 10 minutes' standing is required for full development of the colour, but difficulties due to clouding arise if the standing is prolonged beyond about 20 minutes.

A seldom used combination of the vacuum fusion and chemical distillation methods that records both combined and uncombined nitrogen, consists in absorbing the gaseous nitrogen in calcium vapour, decomposing the calcium nitride in mineral acid to form an ammonium salt, collecting the ammonia conventionally by distillation and determining it by titration.

For an excellent bibliography of methods for nitrogen published before 1935 see G. C. Lloyd's contribution in the Sixth Report on the Heterogeneity of Steel Ingots: Iron and Steel Inst., 1935 (Special Report No. 9, p. 121.)

The third main type of procedure—that of following up the mineral acid conversion of the nitrogen into an ammonium salt directly with colorimetric measurement—has taken several forms. Gotta and Seehof (Zeit, anal. Chem., 1942, 124, 216; Iron Steel Inst., 1943, Translation No. 162) as well as Newell (J. Iron Steel Inst., 1945), have devised direct Nesslerisation methods. In the former, an aliquot portion of a bisulphate fusion extract is Nesslerised after treatment with excess of caustic soda. In the latter, stabilising colloids are used. G. J. Veinberg (Zavod Lab., 1938, **7**, 1,251; ibid, 1940, **9**, 1,073) has evolved an extremely rapid oxidation method based on the formation of a blue colour when solutions of ammonium salts are treated with thymol and sodium hypobromite.

Fig. 3.—Distillation apparatus for determination of nitrogen in iron and steel.



Regarding the origin of this interesting process, I. M. Kolthof and V. A. Stenger devised a volumetric method for determining ammonia by titration with standard hypochlorite solution, the endpoint being a permanent yellow colour, after treating the neutral test solution with potassium bromide and sodium carbonate. Another investigator, Fue-Chine-Schi, had found that albuminous substances oxidized by hypobromite in the presence of an excess of phenols gave a variety of dyes, readily soluble in xylol and ether.

Veinberg coupled these findings and applied them to the determination of nitrogen in ferrous metals, preferring hypobromite to hypochlorite, and L. Lapin and W. Hein have shown that his process is not subject to interference from Ca, Al, Cr, Fe, Co, Ni, Mn, Cu, Cd,

nor from various other cations. Sulphates are without effect. When applied to pure ammonium chloride containing 26·18% of nitrogen, the average yield was 26·0%. The coloured compound is extracted with iso-propyl-ether and compared with the intensities obtained on identically treating standard steels or irons of known nitrogen content. It is necessary to keep the temperature of the solutions below 10° C. The sensitivity of the reaction ($0{\cdot}5 \times 10^{-7}$) g. ammonia per 5 ml. is even better than that of Nessler's reaction (1×10^{-6}) g. per 5 ml. The use of standards of ammonium salt solutions alone in the absence of iron is not permissible, since very different tints are obtained. While not possessing the accuracy of the distillation and vacuum fusion procedures, the method is useful where combination of rapidity and reasonable precision is required.

Method I

Apparatus.—Modified Jones and Morgan Apparatus (Iron and Steel Inst. Carnegie Scholarship Memoirs, 1932, 21, 39). See Fig. 3.

For decomposition of the sample, a 250 ml. round-bottomed flask equipped by a ground joint with a vertical 18-in. reflux air condenser.

For distillation of the ammonia, a 750-ml. distillation flask equipped *via* a ground joint (A) with a top funnel and carrying a bulbular air-condenser side arm (B) connected by a ground or cork joint with the cooling part of a water condenser fitted by a ground or cork joint at the distillate end with an adaptor tube, having a 100-ml. safety bulb, dipping into a 500-ml. conical receiving flask (C). The ground joints are kept free from grease; water provides an adequate seal.

All apparatus is rinsed with nitrogen-free water before use, the distillation apparatus being finally cleaned by passing uncondensed steam for a few minutes before connecting the water condenser with the water supply.

Reagents (exposure to the air is avoided).—Nitrogen-free water (used throughout, containing no ammonia, no nitrate and no nitrite). Prepared in a conductivity still or by digesting distilled water containing 2 g. of Devarda's alloy and 2 g. of sodium hydroxide per litre, for several hours and re-distilling, with rejection of the first 100 ml. of distillate.

Potassium bisulphate (Analar).

Sulphuric acid 1 : 4 prepared by diluting conc. acid (shown by the brucine test to be of low nitrate content) with nitrogen-free water.

Sodium hydroxide, 20% solution of the stick form, free from nitrites and nitrates, in nitrogen-free water.

Hydrochloric acid : $\frac{N}{200}$ prepared by diluting 50 ml. of $\frac{N}{10}$ acid tc 1 litre with nitrogen-free water; 1 ml.= 0·00007 g. of nitrogen.

Mixed indicator. Prepared by dissolving 0·125 g. of methyl-red and 0·083 g. of methylene blue in 100 ml. of absolute alcohol by prolonged digestion at 60° C. until completely dissolved.

Ammonium chloride; standard solution; 0·0382 g. of the Analar salt per litre, using nitrogen-free water; 1 ml. = 0·00001 g. of nitrogen.

Nessler reagent: Prepared by dissolving 35 g. of potassium iodide, and 12·5 g. of mercuric chloride in 200 ml. of nitrogen-free water and adding, with constant stirring, a cold saturated solution of mercuric chloride until a slight red precipitate remains, followed by a solution of 120 g. of sodium hydroxide in 120 ml. of water, finally adding a little saturated mercuric chloride solution, diluting to 1 litre with nitrogen-free water, and transferring to a dark bottle. The solution is allowed to stand for several hours and filtered immediately before use.

Time Required:—2 hours.

Principle.—Solution of the sample in sulphuric acid, whereby nearly all the nitrogen is converted to ammonium sulphate; distillation of the ammonia from boiling alkali solution which converts any undissolved nitrides, and finally volumetric or colorimetric measurement.

Procedure (accuracy ± 0·001%).—A blank determination also is carried out by applying the procedure in entirety in the absence of the sample, or alternatively to a standard steel or iron of known nitrogen content. In routine work there should be a blank for about every six tests.

To the 250-ml. decomposition flask, transfer 1 g. of the washed and degreased sample and 2 g. of potassium bisulphate (Analar.). Alloy samples, especially, should be either in the unannealed or hardened condition; the annealed condition is unsuitable. Add 25 ml. of 1 : 4 sulphuric acid and, using a cover glass, heat almost to the point of fuming. Insert the reflux condenser and heat for at least 15 minutes after fuming commences. Cool, and dilute with 30 ml. of water. Where the silicon content exceeds 1% add 0·1 g. of sodium fluoride for each 1% of silicon present, boil, digest and cool.

Meanwhile, pour into the 750-ml. distillation flask 75 ml of 20% sodium hydroxide solution and 400 ml. of water, introduce a few small pieces of glass tube or silica brick, and distil over a small broad mushroom-type flame or electric-radiation heater until two successive 35-ml. portions of distillate show no reaction with Nessler reagent. At this stage the volume undistilled is usually a little over 300 ml.

Transfer the acid solution of the sample to the tap-funnel and, at a rate sufficiently slow to maintain the boiling, run it into the boiling alkali, rinsing in with small additions of water. Continue the distillation for about 45 minutes, collecting 150–200 ml. of distillate. (In the special case of the nitrogen content exceeding 0·1% extend the distillation for an additional 20 minutes). Complete by one of the two following procedures.

Colorimetric finish.—Specially applicable where the nitrogen content does not exceed about 0·005% nor the carbon content about 0·25%.

Dilute the distillate to 200 ml., add 3 ml. of Nessler reagent, allow to stand for 10 minutes and measure the intensity of colour in a photoelectric colorimeter. Alternatively at the time of adding the Nessler reagent prepare three standards by diluting respectively 1 ml., 3 ml. and 5 ml. of standard ammonium chloride solution to 200 ml., add to each 3 ml. of Nessler reagent, select that corresponding most closely to the test, allow both solutions to stand for 10 minutes, and equalise the intensities by dilution of the more intense. Calculate the nitrogen content of the test by simple proportion, deduct the blank yield and multiply by 100 to give the percentage of nitrogen in the sample.

Volumetric finish.—Specially applicable where the content exceeds about 0·005% of nitrogen).

Add to the distillate four drops of the mixed indicator and titrate with the $\frac{N}{200}$ hydrochloric acid. Deduct the blank yield. Then :

1 ml. of $\frac{N}{200}$ hydrochloric acid = 0·00007 g. of nitrogen
= 0·007% of nitrogen

Method II

Reagents.—Thymol : a 25% alcoholic solution. Sodium hypobromite solution : prepared by stirring 20 ml. of bromine into 100 ml. of a 30% solution of sodium hydroxide in nitrogen-free water and adding a further 100 ml. of the 30% alkali.

Iso-propyl-ether.

Sulphuric acid (1 : 4), prepared with nitrogen-free water.

Standard ammonium chloride solution, prepared by dissolving 0·191 of the Analar salt in nitrogen-free water and diluting to 1 litre. (1ml.=0·00005g.N).

Time Required :—40 minutes.

Principle.—Conversion to an ammonium salt which is oxidized with alkali hypobromite in the presence of a phenol, ethereal extraction of the coloured chinonimin compound so formed, and comparison with standard colourations.

Procedure.—(Accuracy ± 0·002%).

Transfer 1 g. of sample to a small flask equipped with a Bunsen-type valve, add 1 g. of potassium bisulphate and 15 ml. of 1 : 4 sulphuric acid. Heat for 15 minutes after fuming commences. Cool, dilute with 50 ml. of nitrogen-free water, boil, cool and dilute to exactly 100 ml.

Transfer a 10 ml. portion to a boiling tube, cool to below 10° C., by immersing in ice-cold water and shake vigorously with 4 ml. of the 25% alcoholic solution of thymol, and then with 8 ml. of freshly prepared sodium hypobromite solution, cooling to below 10° C. between additions.

After about 2 minutes, add 5 ml. of iso-propyl-ether, shake vigorously and allow to settle. Visually compare the colour-intensity of the ethereal layer with a series of standards prepared either by treating standard steels or irons identically or by treating a single low nitrogen standard steel or iron and adding progressive amounts of standard ammonium chloride to a series of 10 ml. aliquots.

If a photoelectric colorimeter is available, a 2·5 ml. portion may be drawn off from the ethereal layer, transferred to a suitable cell, and the result obtained by reference to a more or less permanent calibration chart.

Theory of Methods—Method I

Uncombined nitrogen sometimes exists in iron and steel, but the amounts are so minute as to defy measurement. The element exists almost entirely in combination. A small proportion may be present in the form of cyanides. With these minor exceptions, the nitrogen is present in the form of nitrides of the constituents. These nitrides have the formulae :

AlN, BeN, BN, CrN, Fe_4N, Mn_3N_2, Mo_2N_2, NbN, P_3N_5, Se_4N_4, Si_3N_4, TaN, TiN, VN, W_2N_3 and ZrN. The elements sulphur, copper, cobalt, nickel and tin are not known to form nitrides in iron and steel.

The nitrides are predominantly those of iron and manganese, which would be completely soluble even in hydrochloric acid ; they are readily decomposed by the sulphuric acid :

$Fe_4N + 5H_2SO_4 \rightarrow (NH_4)_2SO_4 + 4FeSO_4 + 2(H)$

$Mn_3N_2 + 4H_2SO_4 \rightarrow (NH_4)_2SO_4 + 3MnSO_4$

The most stable and least readily soluble compounds are the non-polar nitrides TiN, VN, ZrN, TaN, BN and NbN. These, together with AlN, CrN and Si_3N_4 and any iron cyanide, are virtually insoluble in hydrochloric acid, but are attacked by the fuming sulphuric acid. The higher the furnace or ladle temperature to which they have been subjected, the more sluggish their decomposition.

In every case, the dissolving nitride is converted to ammonium sulphate. Subsequent digestion in the boiling alkali completes the dissolution of residual nitrides other than Si_3N_4 and ZrN, again with formation of the ammonium radical.

Ground joints provide the maximum protection from contamination with extraneous ammonia. A ground joint between the distillation flask and the tap funnel is essential.

Under normal conditions of steady boiling, the whole of the ammonia from all but very high nitrogen contents is expelled within 30 minutes.

The effect of prolonged annealing in rendering the nitrides practically insoluble in acids and alkali alike may be associated with carbide behaviour, especially since high-carbon contents retard decomposition, even where no annealing has taken place.

Use of the dye methylene blue in conjunction with the methyl-red considerably narrows the responsive *p*H range, so affording an even shaped colour change in passing from alkaline to acid solution.

The coloured compound formed by Nessler's reagent has the formula $NH_2\ Hg_2I_3 \cdot H_2O$. Where development of the colour is allowed to reach completion, the intensity is proportional to the ammonia present.

Method II

When ammonium salts are treated with hypobromites, the following reaction takes place :

$2\,NH_3 + 3NaBrO \rightarrow 3NaBr + 3H_2O, +N_2$

The iron is precipitated as hydroxide by the caustic alkali present in the hypobromite solution.

If a sufficient quantity of some kind of phenol is added to the solution, the nitrogen does not separate but the solution assumes an intense and characteristic blue or greenish colour due to the formation of chinonimin. A very suitable phenolic compound is thymol, $C_6H_3(CH_3)(C_3H_7)OH$.

No matter what the phenol used, the coloured compound may be extracted by ether, or benzole or its homologues, or preferably, iso-propyl-ether.

If the solution is slightly acid the ethereal solution of pigment changes from green to red. This is believed to be due to an intermolecular re-grouping in the structure of the pigment, accompanied, perhaps, by molecular dissociation.

The low-temperature working is designed to prevent the entry of bromine into the ethereal layer. A large excess of thymol is detrimental. It is necessary to avoid oxidation of the ferrous iron, since ferric hydroxide tends to be extracted by the ether.

Magnesium and its Alloys

A Review of Technical Progress During 1947

By R. G. Wilkinson, B.Sc.

(*Deputy Chief Metallurgist, Magnesium Elektron, Ltd.*)

During the year there has been steady progress in the development of new applications for magnesium alloys and the restoration of those which existed before the war. Products incorporating these alloys are contributing to the export drive. The main factor has been the establishment of suitable ingot production and foundry technique for commercial casting alloys to meet the need for increased production of castings at competitive prices. Wrought alloys continue to comprise a minor proportion of the total output, but the development of new high strength alloys will increase applications involving wrought products. Further notable advances have been made in research and development, particularly in the field of cast and wrought zirconium-containing alloys. This review indicates that the magnesium industry has laid a firm foundation on which to build steadily and progressively.

Forging

The field of magnesium alloy forging is one which, in recent times, has been given much more attention in the U.S.A. and Germany than in this country, and it is probably true to say that forged components are being more extensively used on the other side of the Atlantic. As is well known, the Germans developed the use of forgings to a very advanced stage prior to and during the war. Large scale trials in the forging of zirconium alloys have indicated that there would be significant advantages attaching to their introduction in place of the aluminium-containing alloys Elektron AZM and AZ855. At a time like the present, when there are many one-off prototype jobs, the free forging characteristics of Z3Z should be of particular help. A potentially important development which is now to be tried is the forging of Elektron MCZ. It is too early to forecast the results, but it seems possible that forgings in this material might find important applications in jet engines where their elevated temperature behaviour would prove of advantage.

Engineering Fabrication

Great strides in technique for the fabrication of wrought magnesium alloys into finished products were made during the war both here and in the U.S.A., particularly in forming and welding. In this country, Cross[15] has summarised his accumulated experience in the manufacture of aircraft fuel tanks, and there have been a number of articles relating to American practice.

*Continued from December issue, page 90.

Welding

No spectacular advances have been made in welding technique during the past year, but much steady development work has been carried out by various companies and research establishments.

In gas welding, it is hoped that the future will see the more extensive use of the comparatively new non-corrosive fluoride base flux of a composition developed independently here and in Germany during the war. The high melting point of this flux renders it unsuitable for use with those magnesium alloys containing appreciable proportions of aluminium and zinc (e.g., Elektron AZM), but it is satisfactory for application in the welding of the binary alloy containing 1·5% of manganese (e.g., Elektron AM503). Using this flux, it is unnecessary to clean the welds (other than for the sake of appearance), since the residues do not attack the magnesium : it is thus possible to make complex welded joints which have hitherto been regarded as dangerous on account of the impracticability of removing flux inclusions.

Advances have been made towards the refinement and extended application of argonarc welding, particularly as applied to heavy sections for structural purposes. In this connection, a heavier torch incorporating water cooling is being developed.

Much work has also been devoted to the argonarc welding of the new zirconium

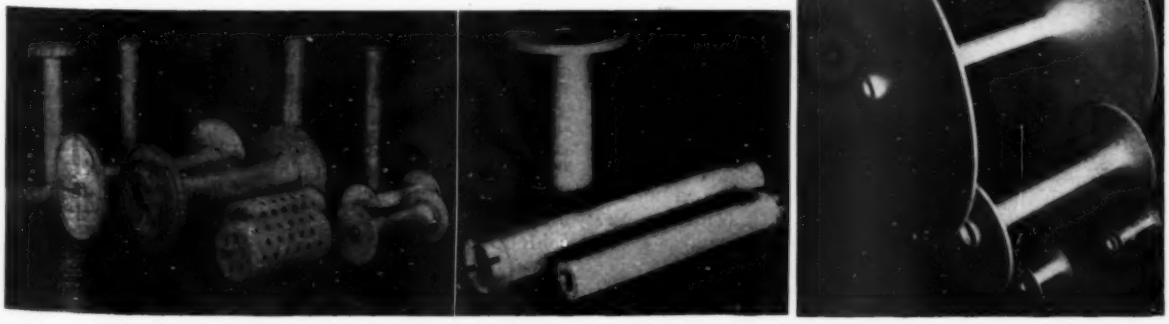


Fig. 12.—Elektron bobbins used in the textile industry.

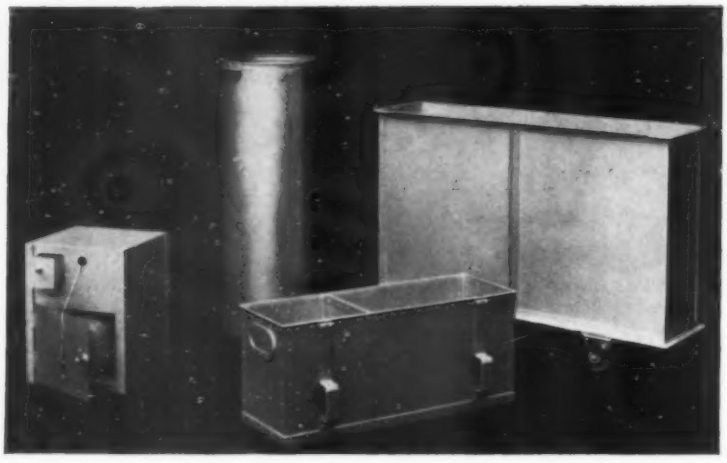


Fig. 13.—Portable containers for the textile industry, fabricated from Elektron sheet and extruded sections.

containing alloys in various forms. The process has continued to be widely used in America, and its production applications have been summarised in a general article by Herbst.[16] It is interesting to note that, whereas the Americans originally used helium as a gas shield, they have now discovered a number of advantages attaching to the employment of argon, used of economic necessity in this country from the beginning. The British Welding Research Association has published a useful booklet[17] in which the four papers on argonarc welding which were presented to the 1946 Symposium are collected together.

There is now a distinct trend towards the more extensive use of spot welding for magnesium alloys, and two valuable papers have been published in the U.S.A. by Hess and his collaborators.[18]

Forming

Little new information of outstanding importance has come to light in the year's literature relating to deep drawing and forming of magnesium alloy sheet, but useful summaries of deep drawing practice in the U.SA. have been written by Gillespie[19] in articles which quote and illustrate a number of interesting applications. Methods said to produce improved cold-forming characteristics have been described in a paper by Carapella and Shaw.[20] Recent work has shown the strong sheet and extrusion alloy Elektron Z3Z to have good formability over an exceptionally wide temperature range—even at temperatures as low as 200°–250° C. This alloy has also been found well suited to spinning.

Corrosion and Protection

Research on corrosion resistance has continued to occupy the attention of a number of workers. Fox,[21] and Fox and Davies,[22] have published papers, one relating to the effect of zinc content on the corrosion resistance of aluminium-containing magnesium alloys, and the other dealing with the corrosion of both normal and high purity alloys in contact with other metals. Bushrod[23] has published a short mathematical treatment of Hanawalt's theory of corrosion, and Jones and Petch[24] have given an account of variation in corrosion resistance over two magnesium alloy sheets. Rogers, Tetu and Livingstone[25] have tested corrosion resistance under various accelerated atmospheric conditions; Ferko[26] has determined the effects on corrosion resistance of various heavy metal inserts and surface treatments, as assessed by arbitrary examination after salt spray exposure; and Rogers and Dingley[27] have investigated the effects of small additions of silver and lead to pure magnesium and casting alloys.

Although intensive efforts are being continued to improve them still further, it is true to say that there is now a growing trend to view the corrosion characteristics of present-day magnesium alloys in reasonable perspective. The corrosion "bogey," at one time so prevalent, is now realised to represent a gross exaggeration as applied to

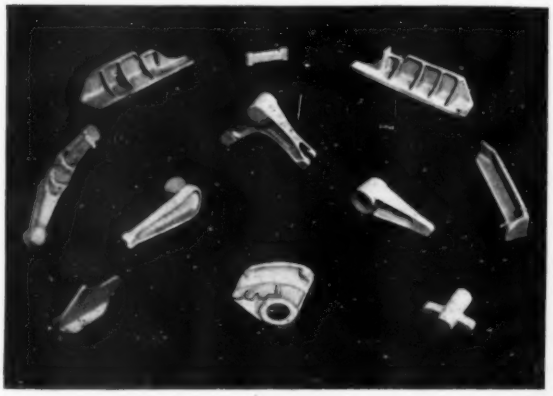


Fig. 14.—Elektron Z5Z castings for aircraft service.

modern products under normal conditions, and it is recognised that current alloys are more resistant to attack than, for example, mild steel. German[28] experience during the war in extensive service applications of magnesium alloys was most favourable. Corrosion troubles were, in fact, so little encountered that it was not considered necessary to manufacture high-purity alloys. At the same time, however, it should not be forgotten that tropical and marine conditions represent special cases calling for the best protection and especially high purity. In this connection, it is not without interest to record that the zirconium alloys show a corrosion resistance approaching that of the high-purity aluminium-containing alloys, and somewhat superior to that of the 1·5% manganese binary.

With the more extensive use of formed and welded high-strength sheet and extrusions in aircraft and other structures, increased attention has recently been paid to the stress corrosion characteristics of such materials. In the short term tests so far carried out at M.E.L., Elektron Z3Z has shown itself to be particularly free from stress corrosion troubles, and to represent a great improvement over the older high-strength aluminium-containing alloys.

A new protective treatment,[29] termed the "Chrome-Manganese" film, has been developed for the zirconium alloys, which is also applicable to the older compositions. This can be applied by immersion in a cold, warm or

hot bath, the immersion period decreasing with increasing temperature. The bath contains manganese sulphate, magnesium sulphate and sodium or potassium di-chromate and has been proved to be particularly economical, whilst producing an attractive black finish having good properties as a paint base.

In the U.S.A., a useful summary of methods of finishing magnesium has been published by Wood;[30] the "Manodyz" process for the anodising of magnesium alloys has been described and illustrated in papers by Gardner Foulke[31] and Black,[32] and Black[33] has also described interesting experiments in the electrolytic polishing of magnesium.

There has been renewed interest in the use of magnesium alloys in photo-engraving, and concise reviews of the various methods have been published by Swayze[34] and an anonymous author.[35]

Sustained attention has also been paid to the use of magnesium alloys in the anodic protection [36 37] of pipe lines, etc., and many experimental data have now been accumulated.

Another special electro-chemical application which has been proposed for magnesium is that of primary batteries, [38 39 40] and research continues on this subject.

Design

Interesting papers have continued to appear in which the stress-strain characteristics of magnesium alloys (and other structural metals) have been related to engineering design. Gill[41] has published an extension of the earlier work of Beilschmidt and Cozzone on the bending strength of materials having non-linear stress-strain curves, and he has paid particular attention to the shifting of the neutral axis such as occurs in magnesium alloys. Van den Broek[42] has included magnesium alloys in a paper on the assessment of aircraft materials from the design angle, and Panlilio[43] has considered the application of limit design to magnesium (and aluminium) alloy structures. (Students of these aspects of design will find that somewhat similar ground has been considered by workers on the problem of brittle fracture in mild steel plates).[44]

Cunningham and his co-workers[45] have carried out an investigation of the plastic flow of a magnesium alloy under biaxial stresses.

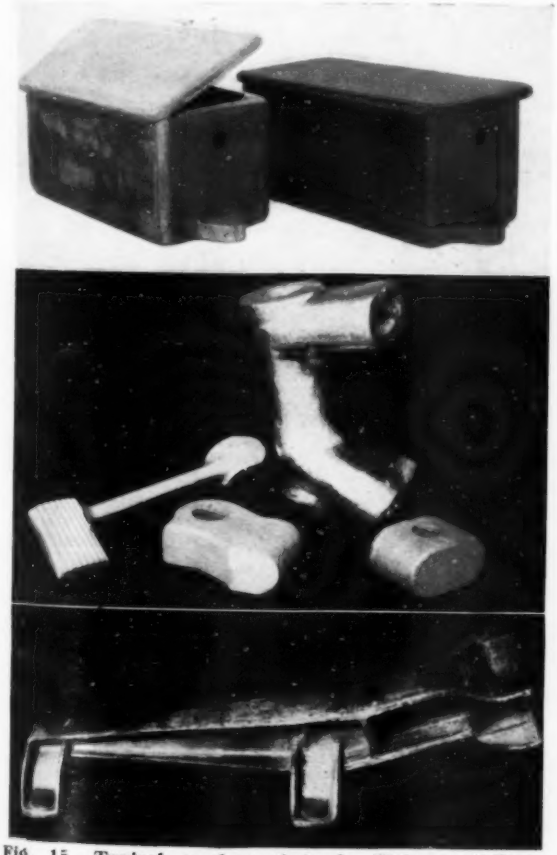


Fig. 15.—Typical sand castings in Elektron "Commercial" alloy. Top: Control box; middle: motor-cycle parts; bottom: structural casting used in an electrical vibrator feeder.

Research

The number of papers published on alloy and similar research in the magnesium field has been rather small. Perhaps one of the most useful contributions in the U.K. has been a paper by Dineen and Benson[46] dealing with mechanical tests, at room and low temperatures, of a number of cast and extruded alloys. (Some other sub-zero test data,[47] in this case for sheet, have been briefly reported in the U.S.A.). Work by Fox[48] on thorium additions to magnesium alloys should also be mentioned.

McDonald[49] has summarised recent alloy and metallographic research carried out by the Dow Co., and George[50] has presented a paper dealing with interesting new metallographic techniques developed by that company.

A Russian paper which has recently become available is that by Kuznetsov,[51] dealing with an X-ray study of the solid solution of aluminium and silver in magnesium.

A research on the plastic deformation of pure magnesium has been carried out by Carapella and Shaw,[52] using the ball indentation methods proposed by Meyer and Hargreaves. Barrett and Haller[53] have written an account of an investigation on twinning, and the same authors[54] have also published results of a study of precipitation in a commercial magnesium alloy in sheet form.

Finally, a large number of war-time research reports[55] of the I.G. Farbenindustrie have become available.

War-time Developments in Germany

A fairly large number of official reports [28 56–64] on magnesium alloy practice and developments in Germany have now been published and these provide a clear picture, the most significant features in which have been summarised in a recent article by Fox.[65] As in this country, the German war-time magnesium situation was governed by shortage: many promising applications, particularly in the wrought field, were replaced by other materials, but, nevertheless, magnesium alloys were still used more extensively in aircraft, and particularly in ordnance equipment, than was the case here.

Whilst many interesting and valuable advances were made in most fields of magnesium technology in Germany, little or no work was carried out in continued development of zirconium or other new alloys.

Applications

Reference has already been made to a number of current applications of magnesium alloys in the U.K., and it will have been gathered that the main fields of usage which can be clearly defined are still the aircraft,

aircraft engine and commercial vehicle industries, with the comparatively recent additions of the textile and printing trades. The list of miscellaneous applications continues to grow, however, and this includes new items for pressure die-castings. In addition to those already shown, a selection of established and new applications are illustrated in Figs. 12–15.

Four general papers, by Hanawalt,[13] Oldt,[66] Eastwood[67] and Fox,[68] have appeared during the year, dealing with the broader aspects of the application of magnesium alloys.

There is an increasing demand in both the U.S.A. and the U.K. to lighten military equipment as much as possible, and so to render it capable of being air-borne; two articles on this subject have been published by Corey[69] and Ghaswala.[70] Military equipment would, of course, absorb relatively large quantities of welded and riveted wrought products, which have already been proved capable of withstanding the heavy shock loadings involved in service, for example, in a 105 mm. Howitzer carriage.

A significant application of spot-welded magnesium sheet in control surfaces of aircraft has been described by Saunders.[71] His company, the Beech Aircraft Corporation, found many advantages, including greater strength, fewer parts, less weight, lower tooling costs and a worthwhile saving in labour. It has also been found economic in the U.S.A. to use magnesium alloy sheet for the bodies of delivery vehicles which must be both light and of large capacity – e.g., bakery vans.[72]

It should be mentioned here that the old fear of insuperable fire hazards in the employment of magnesium alloys, now practically extinct, received something approaching a death-blow in some tests carried out by the Admiralty over a year ago, the results of which have been published.[73]

As a final remark, it can be said that the magnesium industry, after many difficulties following the war, has now laid a firm foundation upon which to build steadily and progressively in the future.

Acknowledgments

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Metallurgy and Atomic Energy

By C. Hubert Plant, F.I.M.

There can be no doubt that the advent of atomic energy will become a landmark from which to survey future developments in all fields; it is an event of tremendous implications and few will yet be able to assess the effect of its impact on human affairs and upon industry. Various aspects of the subject from a metallurgical point of view will be discussed by the author in a short series of articles. In the present article, the second of the series, attention is directed to a consideration of the nucleus of the atom.

So far only the negative charges or electrons surrounding all atoms of matter have been considered in any detail and efforts made to indicate how the universal phenomenon of energy enters into their mechanism, but it is necessary to devote considerable attention to the nucleus, because it is almost solely with this portion of the atom that the most recent knowledge regarding atomic energy is concerned.

It has been stated that the nucleus contains practically the whole of the mass of the atom within itself. The mass of the electron is only 1/1840th the mass of the hydrogen atom so that, even in those elements possessing the greatest number of electrons, their combined mass is a very small proportion of the mass of the atom.

Before proceeding further, however, it will be useful to obtain a clear understanding of what is meant by the word "mass." There is often confusion between "weight" and "mass." Weight, of course, is a measure of the gravitational pull on a certain mass, and varies in accordance with varying gravitation. For instance, a so-called mass of 6 lb. will, at the surface of the earth weigh approximately 6 lb., but on the surface of the moon it would only weigh 1 lb. At one time it was thought that the mass of any matter was a constant measure of that matter and was defined as being a measure of the quantity of matter. This is not correct. Mass is actually a measure of the effort required to set a body in motion or if in motion, of the effort necessary to cause it to alter its path. The more difficult it is to move a body, the greater is its mass and the more correct definition of mass is, therefore, that it is the measure of the inertia of matter. But the problem is still a little more complex than this. Supposing a piece of matter is travelling at a certain speed, and it is desired to double that speed. Common sense tells us that it will be more difficult to cause an alteration, say in direction, at the higher speed than at the lower. Sir J. J. Thomson, from purely theoretical considerations, showed that the mass of an electrified body should vary with its speed and this theory was quickly proved by experiment. Einstein, studying the same subject carried it further. He postulated that this mass would continue to increase with increasing speed until, at the speed of light, it would become infinite. Furthermore, he stated that increase in speed produced increase in energy which is merely the classical theory of kinetic energy, where the energy is equal to half the mass multiplied by the square of the velocity. But he went still further and showed that every form of energy has an equivalent mass. This is now one of the fundamental laws of the universe and is of the utmost importance. Thus, it must be realised that bodies may have differing masses under differing conditions. It is perhaps somewhat difficult to realise that the mass of a train travelling at 60 m.p.h. is greater than it was when standing at the platform of a station but it is nevertheless true.

Having attempted to clear up the point about mass, it is possible to revert to a consideration of the nucleus of the atom.

The Nucleus of the Atom

As previously mentioned, Sir J. J. Thomson had no idea of the existence of the weighty positively charged nucleus of the atom. He had considered the electrons to rotate within a sphere of positive electrification and it was for Rutherford and his co-workers to discover the true facts. It was brought about by Rutherford's concentrated work on radio-activity and, in particular, in connection with alpha particles. As is well known, radioactive substances emit three general classes of radiation, which have been termed the alpha, beta and gamma rays. The beta variety are nothing more than electrons, moving with varying velocities, some nearly as great as that of light. If these beta rays are fired at metallic atoms in the form of thin sheet or foil it is found that they will pass through the foil, but that their paths on emergence have diverged due to their repulsion by the electrons forming the planetry orbits of the atoms. This divergence can be determined and from it, by a very simple piece of mathematical reasoning, it can be shown that the electrons in the bombarded metal form a very small proportion of the mass of the atom. The conclusion can, therefore, be drawn that the greater part of the mass is located in the positive portion of the atom.

The alpha rays from radioactive substances are positively charged particles and are, in fact, helium nuclei consisting of two protons and two neutrons. This being the case, its mass is of the order of 7,000 times the mass of the electron and, if these particles are fired at metallic substances in the same manner as the beta particles, they would obviously not be turned aside by the much lighter electrons of the atoms. Yet when experimenting in this manner, Rutherford and his colleagues found that, not only were they deflected from their course through quite large angles, but, in some cases, they were completely turned back. Rutherford was able to show quite definitely that the cause of this deflection lay in the interior of the metal, and, as the deflection could be so serious, the cause of it must also be something of the same order as the alpha particle, so far as mass and charge were concerned.

The inference which Rutherford drew was that if the sphere of positive electrification of Thomson were concentrated into an extremely small nucleus, the

power necessary for the deflections was there. So far, he had come to this conclusion by purely mathematical reasoning, but he devised an ingenious experiment and was able to confirm his theories by entirely accurate, quantitative tests, as well as by mathematical reasoning. And so the nucleus was born, or at any rate discovered. At first Rutherford spoke of it as the " central positive charge " but quickly replaced this cumbersome appellation by the more exact word " nucleus." Also, in those early days it was assumed that it consisted of positive charges or protons, together with electrons, the net positive charge balancing the number of electrons in the outer orbits. The neutron had not then been thought about, but Rutherford had realised that there were enormous forces locked up within that nucleus and it is those forces which are to-day described generally under the term, Atomic Energy.

Following the discovery of the nucleus came the work of Bohr and Moseley, already mentioned and then the first World War, which postponed for a long time the wonderful work which was to come. It was, however, in 1914 that the first hint of what was to follow emanated from Rutherford's laboratory at Manchester. This was the discovery made by Marsden that if alpha particles were projected into hydrogen gas, some of the hydrogen atoms were driven forward by the heavier helium nuclei in the same manner that one marble is pushed forward by another, and it is very probable that this discovery gave Rutherford the first ideas which later paved the way for his great transmutation experiments.

After the end of the War in 1918, which was Rutherford's last year at Manchester, work was again continued upon the nucleus of the atom and it was in the Philisophical Magazine, volume 37, in early 1919 that he first described the possible artificial disintegration of nitrogen. This statement was of such a revolutionary character that before the suggestion could be considered a fact, a considerable amount of careful experimental work had to be undertaken. Rutherford was now at the Cavendish Laboratory in Cambridge and in June, 1919, he was able to say with certainty that alpha particles, bombarding nitrogen gas had driven from the nuclei of its atom, positively charged particles which were to all intents and purposes exactly the same as the positive nucleus of the hydrogen atom. It was not, however, until a British Association Meeting in Cardiff in 1920 that Rutherford gave the name " proton " to this particle, which name it has held ever since. Work continued by bombarding atoms of many of the lighter elements with alpha particles, from the atoms of which protons could be ejected. But it was not until his lecture before the Royal Institution in April, 1925, that Rutherford was able to startle, not only the scientific world but the world at large by stating that one atom had been transmuted into another, heavier than the original one. It was the original experiment of bombarding nitrogen by alpha particles, but in this instance P. M. S. Blackett, of the Cavendish Laboratory had succeeded in proving by photograph that when the alpha particle drove the proton from the nitrogen atom, it acted much in the manner of a cuckoo. That is, it threw out an egg in the shape of a proton and stuck inside the cosy nest of the nitrogen atom. The implications of this are so important in relation to what is to follow that it is necessary to consider what actually happened in some detail.

As has been explained, the alpha particle has an atomic mass of four, while the hydrogen nucleus or proton has a mass of one. If, therefore, the nitrogen loses a proton, its net atomic mass will be one less, but by gaining the alpha particle the resulting mass will be increased by three. Again, the atomic number of the alpha particle is two and of hydrogen one. By losing the proton and gaining the alpha particle, therefore, the net increase in charge on the nitrogen is one. The resulting atom is accordingly one with an atomic mass of 17 with a charge of 8. Now the gas oxygen has a net positive charge of 8 with a mass of 16, but there is an isotope of oxygen with a mass of 17, and it is this isotope into which the nitrogen atom had been transmuted. This reaction can be illustrated by means of symbols in the equation form as follows :

$$N_{7}^{14} + He_{2}^{4} \longrightarrow O_{8}^{17} + H_{1}^{1}$$

Here the number placed at the top of the chemical symbol represents the atomic weight, or more correctly the atomic mass, while the number at the foot of the chemical symbol represents the net charge on the atom. As we shall make clear later the charge number gives the number of planetry electrons, hence, the number of protons in the nucleus to maintain the charge balance, while the difference between the two numbers indicates the number of neutrons in the nucleus.

Which is the cue for the entrance of the next actor in this atomic energy drama—namely, the neutron. It was in 1920 that Rutherford had, in the course of one of his lectures, hinted that he considered there should be a particle carrying no charge but of more or less the same mass as the proton and subsequently, although there had been evidence that such a particle existed, no one had been able to identify it. It is not within the scope of these articles to trace how this was done, but the final evidence which enabled Chadwick at Cambridge in 1932 to clinch the matter was supplied by Professor Joliot and his wife, Irene Curie, daughter of the Curies of radium fame. The fact was established that there is such a particle as Rutherford had foreshadowed in 1920. It carried no charge and its mass is almost identical with that of the proton.

The advantages to be gained by the use of this particle as a projectile for bombarding atomic nuclei were at once apparent. Hitherto the particles used for the transmutation experiments had always been either protons or alpha particles, but it was always realised that there were great disadvantages in the use of these agents. In the first place they are both positively charged, consequently under all ordinary conditions they are repelled by the positively charged nucleus of an atom. On the other hand, there is a certain amount of attraction for them by the negatively charged electron clouds surrounding each atom, which apart from all other considerations tends to slow them down before they have had a chance of colliding with a nucleus. To overcome these difficulties means were found to speed up the protons or alpha particles so that their velocity was sufficient to overcome the braking effect of electrons and was sufficient also to overcome the repelling force of a nucleus. But with the advent of the neutron, both these difficulties at once disappeared. The neutron is of no interest to an electron cloud, neither has the nucleus any feeling of repulsion for it, consequently the neutron can wander where it pleases or where it is sent. Furthermore, this particle can be accelerated and any required speed given to it. Another point in favour of the neutron was that it at once simplified the whole problem of the

structure of the nucleus. As has been pointed out, it was thought that nuclei consisted of protons and electrons, but it was quickly realised that the secret of the nucleus was that it consisted of protons plus neutrons to produce the mass and the necessary net positive charge.

As to what the neutron actually is has not yet been completely explained. It has been suggested that it is a close union between a proton and an electron, but in this case its mass must of necessity be greater than the mass of the proton by 1840/th which does not agree with observed facts. Alternatively, it may be a fundamental particle and a proton may be a union between this and a positron. A positron, it should be mentioned, is an electron bearing a positive instead of a negative charge and has been identified, but the same argument applies against this supposition. A third theory, which seems to be most probable, is that a neutron is merely a proton which has lost its positive charge. For the present purpose, however, the matter is not of intense importance and need not be considered further.

Physicists were quick to realise the importance and usefulness of the neutron for nuclear bombardment and some surprising facts were discovered. These will be dealt with in some detail a little later and in some respects may almost be described as metallurgy gone crazy.

Mention has been made of the fact that when protons or alpha particles were used for bombarding nuclei, their velocity was considerably accelerated. This was done by subjecting the bombarding particles to an intense electric field either by means of an electrostatic generator or by means of an electro-magnetic machine termed the cyclotron. A particle of 500,000 electron volts actually means that the particle has been accelerated by means of a potential of 500,000 volts and does not mean that the particle itself has this degree of electric tension. It was by accelerating protons in a field of 600,000 volts that two of Rutherford's workers at Cambridge in 1932 made atomic history. When the facts became known, the popular press produced startling headlines about the splitting of the atom and allowed its imagination to soar to astonishing heights. What had actually happened was that protons with an accelerating voltage of over 600,000 volts had been shot at lithium atoms. The protons had entered the lithium nuclei, had been absorbed and then the atoms had split violently into two fragments, each an atom of helium. The action is shown thus :

$$Li_3^7 + H_1^1 \longrightarrow He_2^4 + He_2^4$$

Now this really was a startling result and was the first instance recorded of atomic fission. In view of all that has happened since it is of interest to note that in a lecture to the British Association the following year Lord Rutherford said : " These transformations of the atom are of extraordinary interest to scientists, but we cannot control atomic energy to an extent which would be of any value commercially, and I believe we are not likely ever to be able to do so." It is to be regretted that he did not live to know what is known now. By a curious coincidence it was in the same year that Rutherford made these remarks that another discovery was made in Paris by the Curie-Joliots, which has already proved to be of great benefit to mankind. In continuation of the work on atomic bombardment which was proceeding in nearly every physical laboratory in the scientific world, the Curie-Joiliots were firing alpha particles at boron. This transmuted into an unusual form of nitrogen and threw off a neutron, thus :

$$B_5^{10} + H_2^4 \longrightarrow N_7^{13} + n_0^1$$

This action was nothing out of the ordinary, but then something very extraordinary happened. This nitrogen became radio-active, ejected a positron and became an isotope of carbon in accordance with the following reaction :

$$N_7^{13} \longrightarrow C_6^{13} + p_1^0$$

This effect became known as artificial radio-activity and more will be said of it later.

But yet a third matter of importance came to the front in 1934. This, as will be seen when the energy relations of atomic processes are considered, may become of even more vital importance than the now popular uranium. In 1932 it had been proved that the actual atomic weight of hydrogen was not 1·00 but 1·0081 which suggested a possible isotope of the gas, hitherto undetected. American scientists looked for this isotope and were able to isolate it, of mass number 2. It has been given the name deuterium and the nucleus of this atom has been called the deuteron. It has become known by its connection with " heavy water," that is water containing deuterium instead of ordinary hydrogen. Not only this, but at the Royal Institution Meeting in 1934, Rutherford was also able to announce yet another form of hydrogen of mass number 3 which is termed tritium. It will be seen later that the knowledge of these " heavy " hydrogen atoms may open the door to a source of energy the possibilities of which are staggering.

And now comes the last episode in this amazing history of atomic physics. From hydrogen of mass 1, we turn to the heaviest element, of mass 238, Uranium. It was in the years 1937 and 1938 that Otto Hahn, Lise Meitner, the Curie-Joliots and others on the Continent opened up the tremendous possibilities of atomic fission and chain reaction which ultimately produced the atomic bomb. Fission has already been referred to in the splitting of the lithium atom but true fission was discovered in 1938 when uranium was bombarded by neutrons and the artificially radioactive substance released was shown to be similar to the element barium. Without going into the arguments, this suggested that the uranium had split into two more or less equal portions during which splitting operation large amounts of energy were released.

The magnitude of the energy emitted was predicted to be of the order of 200 million electron-volts. In ordinary electrical units this amount of energy is very small but if, say an ounce of uranium is considered, the number of atoms in that ounce calculated and it is assumed that fission of each atom could be obtained, the resulting energy would be of the order of 200 million units of electricity, enough to reduce the current shortage very materially. How this energy is developed will be discussed soon. But this atomic fission was not the only fact which came to light in these experiments with uranium, the more surprising result being the so-called chain reaction. Everyone is familiar with the popular coconut shy in fair grounds. Let the reader suppose he visits one such place and is prepared to " have a go." Instead, however, of the usual array of coconuts he finds to his amazement that there are hundreds of them and upon paying his sixpence he is given only one ball

and is told by the showman that he will obtain a prize only if he is successful in knocking off every one of the hundreds of coconuts. This appears utterly impossible but, having paid his money, he takes the ball, throws it and succeeds in knocking off one coconut from its stand. To his intense surprise, however, this coconut breaks into two, and from each half other balls spring out and themselves shoot at other coconuts which are in turn knocked off, repeat the halving process and release more balls. This goes on until, in a very short space of time the whole of the coconuts have been knocked away and split up. That is the idea underlying chain reaction. If the coconuts are atoms of uranium and the first ball is a neutron, then there is an example of fission and chain reaction. But all this happens in a fraction of a second, so that the total energy emitted becomes prodigious, so much so that the atomic bomb which was dropped on Hiroshima produced more energy than the power of 20,000 tons of high explosive.

Now it is not the purpose of these articles to describe the atomic bomb, past, present or future as a means of destruction, neither will space admit of technical discussions regarding the various isotopes of uranium used for the purpose or of the critical speed of the neutrons employed and many other matters. Would it were possible for nations to forget that atomic bombs had ever existed because otherwise the possibility of such a weapon is the greatest menace that human civilisation has ever had to face. On the other hand, it is now our duty to discuss the probability of atomic energy being used for the benefit of mankind and to do everything in our power to utilise it for such purposes if this be possible.

To this end it is now necessary to turn to a more critical discussion of the energy capabilities of atomic nuclei. How is that energy derived—from whence does it come, whither does it go? What is the difference between the energy of deuterium and uranium and if both these elements possess energy in enormous quantities, do other elements which have been transmuted possess the same? The remainder of this series will endeavour to answer some of these questions.

To be continued.

The Platinum Metals in 1947

World demand for platinum metals was maintained at a high level during 1947, according to a recent statement by Dr. Charles Engelhard, President of Baker & Co., Inc., with United States consumption estimated at 375,000 ounces including approximately 200,000 ounces platinum and 150,000 ounces palladium. Although the United States is the largest consumer of platinum metals, industrial reconstruction in Europe requires large quantities of these metals.

United States imports during the first ten months of 1947 approximated 260,000 ounces of platinum metals in all forms including 44,500 ounces in unrefined materials, mostly from Colombia. Imports of refined platinum metals included approximately 45,000 ounces of platinum and 63,000 ounces of palladium from Canada, 32,500 ounces of platinum and 36,500 ounces of palladium from Russia, and 18,500 ounces of platinum and 4,700 ounces of palladium from the United Kingdom.

Although most people think of platinum and palladium and the other platinum metals in connection with jewellery, a large proportion of the available supply of these precious metals is used for industrial purposes because they reduce costs of production and maintenance. The economic advantage of using platinum metals for critical parts of equipment is well recognised by producers of rayon, glass and electrical instruments, and manufacturers in other fields are giving increasing attention to the money-saving possibilities of platinum metals. The development and perfection of bi-metallic products comprising a base metal faced with platinum has enabled users to employ these precious metals to a better advantage.

The rayon industry continues to be one of platinum's most enthusiastic users. The economy of this metal alloyed with rhodium is demonstrated by its constant use as spinnerets which are utilised in generating rayon fibres used in the manufacture of clothing fabrics, tire 'cord' and many other items. These spinnerets are fine examples of the excellent fabricating qualities of the platinum metals as over 15,000 holes of about two and one-half thousandths of an inch in diameter are not uncommon in a spinneret of two and one-half inches in diameter. Fireproof fabrics such as the new covers for ironing boards are made from glass fibre produced from orifices of platinum metals. In the mass production of electric light bulbs molten glass is fed continuously by feeder dies of platinum metals.

All the platinum metals are used in production of jewelry and other adornments. Alloys of platinum hardened with iridium have been used for a long time in the most expensive type of diamond-set jewellery. During recent years an increasing quantity of jewellery has employed an alloy of 95% palladium and 5% ruthenium. Wedding and engagement rings made of this material have the strength and whiteness desirable for this type of jewellery, and palladium also has lightness to save weight in earrings, brooches and other large pieces. The versatility of palladium has been further demonstrated by the recent development of palladium filled jewelry.

The immediate demand for platinum and other platinum metals shows no signs of slackening. Research and development continue to find new applications for these metals which are assuming an important role in modern industry. Indeed, it is doubtful whether there is a research laboratory of any importance anywhere which does not consider the employment of the platinum metals, particularly in their purest state, to reach that perfection in quality which eventually will lead to, and form the basis of, remarkable developments in industry.

Stamping Laminations

Some interesting dies have been made for stamping laminations out of silicon steel for use in making electric motors. These dies are of Edgar Allen K.9 oil hardening steel, and were made by Messrs. Purfection Tools Limited. They are form ground all over, and measure approximately 10 in. outside diameter, the outside diameter of the component being approximately 4 in. K.9 steel has been used for this type of tool for a considerable period by the firm concerned, being capable of giving the fine ground limits and finish required.

Copper and Copper Alloys*

A Survey of Technical Information During 1947

By E. Voce, Ph.D., M.Sc., F.I.M.

Copper Development Association

Steady and persistent progress has been made in the production and application of copper and copper alloys and in grappling with the problems underlying their behaviour. In this survey it will be seen that developments cover a wide field, including casting and fabricating operations, many aspects concerning properties, corrosion and protection, activity in the powder metallurgy field, joining technique, metal finishing, and physical metallurgy and testing; developments that represent a body of solid achievement.

Corrosion and Protection

Dealing broadly with corrosion, part of a series of articles in *Sheet Metal Industries*[59] provides a useful summary of the corrosion of copper and its alloys, while papers have also appeared on several specific corrosion problems such as those encountered in the oil industry,[60] sugar refineries,[61] tropical service,[62] paper making,[63] and the storage of crude turpentine.[64] For most such purposes copper alloys compared favourably with other materials of similar cost.

Copper and Underground Piping.—In view of the present shortage of lead and the greatly increased use of copper pipes for all forms of plumbing, the investigations of Gilbert[65] on the corrosion of these materials when buried in soil are remarkably opportune. Gilbert used tubes of tough pitch arsenical copper, virgin lead, B.N.F. ternary alloys, and tellurium-lead, buried in seven different soils for periods up to ten years. He expresses the opinion that it would be unwise to bury unprotected pipes of any of the materials in the two most corrosive soils, but states that bare copper pipes could be expected to last indefinitely in all the others. Further, he shows that copper gives superior performance to lead or lead alloys in all the soils with the single exception of wet acid peat, in which some, but not all, of the lead and tellurium-lead specimens were slightly better.

Corrosion of Gas Water Heaters.—Also of interest to plumbers is the work of Booth and his associates[66] on the corrosion of gas water heaters, comprising an investigation of the causes and incidence of the fluffy blue deposit with which all users of gas-operated water heaters are familiar. Heaters with both thick and thin solder coatings were used, and gas of both high and low sulphur content was burned. The heating was intermittent to aggravate condensation of moisture from the burning gas. The main conclusions were as follows:—

(1) A reduction in the sulphur content of town gas from 17 to 3 gr. per 100 cu. ft. reduces the corrosion of water heaters nearly proportionately.

(2) There is probably a limiting value of the sulphur content below which corrosion begins to increase again. The limiting value may lie in the neighbourhood of 3 gr. per 100 cu. ft.

(3) Oxides of nitrogen may play an important part in the mechanism of corrosion, particularly with gases of sulphur contents below this limiting value.

(4) There is less corrosion in heaters with a moderately thick coating of solder of high lead content than in heaters with a thinner coating of higher tin content.

(5) The rate of formation of corrosion deposit increases with time, but probably reaches a limiting value.

(6) A 10% reduction in the thermal input did not result in less corrosion, except in the case of the gas containing 3 gr. of sulphur per 100 cu. ft.

The solder coating is gradually destroyed during service, and the limiting rate of corrosion referred to is presumably attained when it has completely disappeared. It would appear that there is much room for improvement in methods of protecting copper from the hot products of combustion of town gas, and that a profitable field for research is open in this direction.

"Destannification" of Bronzes by Superheated Water.—Clark[67] has examined the attack of super-heated water on the cast phosphor bronze feed pumps. Penetrative attack occurred via the network of tin-rich phase, or perhaps more strictly via the pores present in it. The result was an effect closely resembling the familiar dezincification of alpha-beta brasses, in that the tin-rich phase was removed, its place being taken by "lakes" of redeposited copper surrounded by areas containing oxides, hydroxides, etc., chiefly of tin. Since the attack was found to have taken place in alkaline (treated) feed water, and also in acid (carbon dioxide) condensate, it was inferred that little beyond water is necessary to cause it, though condensate was more corrosive than feed water. Corrosion appeared to be confined to temperatures above 150° C. and porosity, or opening the pores by machining away the cast surface, had a marked accelerating effect. Wagner, Decker and Marsh[68] also investigated the corrosion occurring in boiler feed pumps and regulator valves, and reported that bronzes and Monel offered good resistance. This bears out service experience.

Stress Corrosion of High Tensile Brasses in Sea Water.—Examining the stress corrosion of high tensile brass castings for submarine service, Sheenan and Dickerman[69] found that materials of the alpha-beta structure were less susceptible to failure than those consisting entirely of beta. Stresses somewhat higher than the yield strength were necessary to initiate failure in sea water. These facts are by no means new to those who have made a study of such problems, or who have practical experience with brasses under marine conditions, but it is opportune that they should be reaffirmed.

*Continued from December issue, page 84.

Anti-Fouling Paints.—The contribution which copper has made to the protection of ship's bottoms from fouling by marine organisms is widely recognised. Much activity has recently been expended in the development of anti-fouling paints, particularly by the Marine Corrosion Committee of the Iron and Steel Institute.[70] Most of such paints contain copper or a copper compound which is leached out by the water at a slow but carefully controlled rate, and renders the interface between the water and the paint so toxic that barnacles and other organisms, both animal and vegetable, are deterred from lodging or from breeding there.

Powder Metallurgy

Unusual activity has been displayed in connection with powder metallurgy during the year, and copper ranks second only to iron in importance in this field. A valuable bibliography covering the whole subject has been published by Price[71] while general reviews have been written by Jones[72] and by Tait.[73] Chase[74] has compared fabrication by powder metallurgy with other methods from the economic angle, and describes the factors that must be taken into consideration in discriminating between them. While for many purposes powder metallurgy is essential, this paper will repay study by those who are contemplating its substitution for hot stamping, die casting and similar processes.

Last year, a collection of papers on the mechanism of the pressing and sintering processes appeared under the auspices of the American Institute of Mining and Metallurgical Engineers,[75] but a more dominating position is taken by the recent symposium of the Iron and Steel Institute. Twenty-eight papers were presented and of these no less than sixteen deal either directly or indirectly with copper powders. In the limited space available it is impossible to do adequate justice to this mine of information and no more than a brief outline of certain of the papers can be given.

Miller[76] describes the method employed in the production of both ferrous and non-ferrous metal powders. Copper is normally produced by one of three methods, namely atomisation of liquid copper in a high pressure fluid stream (generally water), reduction of the oxide with hydrogen, or electrolysis. Flake powders, the production of which is described by Meyersberg,[77] are used mainly in the production of paints and ink. They are made by comminution in stamp or ball mills, and differ from the powders employed for the manufacture of sintered articles in that each grain is a minute flat leaf.

Cook and Pugh[78] give detailed attention to the features involved in the pressing and sintering of copper powders while Lennox[79] describes the production of a number of engineering components by powder metallurgy. Because of the evolution of zinc vapour during the sintering process, it is not easy to prepare brass compacts from powdered zinc and copper, but Lennox overcame the difficulty by using 70 : 30 brass powder in the previously alloyed condition.

Copper powders are largely used together with tin in the manufacture of porous bronze bearings, and a paper by Carter and Metcalfe[80] gives a useful description of the processes involved. It was found that the peritectic reaction at approximately 800° C. between the solid solution and liquid is of prime importance in the sintering operation : a much more homogeneous product could be obtained by sintering in the higher temperature range. Tait[81] also pays attention to the production of sintered bearings and thrust washers, especially those of a porous and self-lubricating character. The production of filters from highly porous metal compacts is described by Sinclair.[82] Examples are given of the flow and pressure-drop relationships through porous filters produced from powders, together with micrographs of the pore-size and shape. Practical applications of such filters are discussed in considerable detail, and it is shown that they have a wide range of use. A special application of highly porous sintered bronze, in this case containing nickel, to the de-icing equipment on aircraft is described by Aircraft De-Icing, Ltd.[83] The function of the porous material is to distribute the de-icing fluid over the parts of the aircraft upon which ice would otherwise form to a dangerous degree.

Sintered iron-copper compacts are described by Northcott and Leadbeater,[84] while the operations of pressing and sintering as well as the properties of similar mixtures are given by Chadwick, Broadfield and Pugh.[85] Copper-iron alloys form no brittle phase, and it is possible to obtain strong sintered compacts over a wide range of composition. Almost, but not quite, within the category of powder metallurgy comes a description[86] of the production of bearings from compressed steel-wool impregnated with copper. Such bearings are said to withstand extreme cold and to facilitate proper lubrication through the porosity which is allowed to remain.

Joining

Welding and brazing of copper and its alloys.—A highly practical paper by West[87] gives full manipulative instructions for the autogenous welding of copper and its alloys, while Davis deals similarly with the welding and brazing of copper[88] and of brass.[89] In each case the necessary precautions to be taken are outlined in relation to the basic scientific aspects of the problem, and the papers merit careful study by all who are handling the joining of copper and its alloys. Cook and Davis[90] also review the present position, describing recent advances in the pressure welding of copper and its alloys. They emphasise the deleterious effect of selenium and tellurium on the weldability of copper.

While Rollason and Marsh[91] reported hot cracking in 7% aluminium bronze multi-run seams, ascribing it to the effect of an unknown impurity, Evans and Haim[92] have developed the use of specially coated electrodes of complex aluminium bronze containing iron, nickel and manganese to avoid lack of ductility in the weld metal. Provided that the flux coating of the electrode is carefully dried before use, it is claimed that excellent welds can be made, not only on a basis metal of aluminium bronze but also for repair work on cast iron and steel, and for joining these to aluminium bronze.

Rubber-to-brass bonds.—The bonding of rubber to metals, and especially to brass, has received considerable attention. A comprehensive German review of the subject has recently come to light,[93] and Messenger[94] has given full details of the technique of bonding rubber to steel by means of brass plating ; but a more fundamental contribution is that of Buchan and Rae[95] on the nature of the rubber-to-brass bond. Zinc, copper, sulphur and rubber probably take part in the bond formation, for all four are present at the interface. Sulphur reacts with copper and zinc to form sulphides, and with rubber to give the vulcanised product. Copper and its compounds are notable for their deleterious

effect on rubber, to which they tend to impart a gummy consistency. Zinc compounds, on the other hand, are not harmful, and zinc unites with rubber only with difficulty. In bonding to brass it appears, therefore, that copper is the active agent, while the zinc exerts a measure of control over the reaction.

Electro-plating and Finishing

The technical progress in regard to metal finishing which occurred during the year 1946 is described by Mozer[96] in an article which covers corrosion prevention, polishing, cleaning, pickling, coatings of all kinds, electroforming, the metallising of non-conductors, colouring and testing. No revolutionary developments appear to have taken place. Attention must be drawn to the International Conference on Electrodeposition which was held in London in September, 1947.

Electrolytic polishing.—Though the electrolytic polishing of copper and its alloys has not yet attained industrial importance, there seems little doubt that it represents a promising method of finishing, provided that its limitations are recognised and accepted. It is folly to expect, for instance, that rough surfaces such as those of a casting, can be smoothed by electrolytic polishing, though they can, indeed, be brightened. Moreover, electrolytic polishing can be applied more satisfactorily to homogeneous materials than to those which contain several micrographic constituents. A paper by Lainer[97] deals particularly with the relationship between the concentration of phosphoric acid and current density in the electrolytic polishing of copper and its alloys. He maintains that as the concentration of phosphoric acid in the bath increases, the optimum current density is lowered and the time required for polishing is correspondingly lengthened. While this may be true, experience shows that relatively concentrated solutions give more reliable results. Lainer maintains that the electrolytic polishing of copper and its alloys is commercially practicable. Mazia[98] describes the electrolytic polishing apparatus used at Frankford Arsenal and a brush method of polishing selected areas of heavy objects which are not amenable to mechanical treatment or electrolytic polishing in tanks. Details of the solutions and conditions are given for the polishing of brass in addition to other materials, and it would appear, therefore, that the method is being used for the finishing of certain articles at Frankford Arsenal. Evans and Lloyd[99] have reported work on the electrolytic polishing of nickel-silver and stainless steel spoons. The electrolyte used contained 37% ortho-phosphoric acid, 56% glycerine and 7% water, and for nickel-silvers an operating temperature of between 50–70° C. at a current density of 1 amp/sq. in. was found to give the best results. After electrolytic polishing, the surface of the nickel-silver spoons often showed a distinctly rippled effect. A few plating experiments showed, however, that 0·003 in. of nickel obliterated the rippling.

Other surface finishes.—An entirely different type of surface finish is that given by the "Ebonal" process described by Mayer and Vincent,[100] for the blackening of brass and similar alloys. It was used during the war for the production of a durable dark finish on various types of armament, while the ebonised surface serves as a good base for painting and lacquering.

Somewhat revolutionary experiments have been conducted by Chapping and Hayward[101] on the coating of mild steel with copper-titanium alloys by the application of heat to titanium hydride in the presence of copper, in an inert atmosphere. Such coatings have a high silvery lustre and exceptional hardness, though they appear to be somewhat brittle. They have a dense texture and are probably resistant to corrosion.

The metallising of non-conductors.—With the advances made by plastics, the art of coating them with metals is becoming increasingly important, both for decorative and functional purposes, and a book on the subject by Wein[102] provides a comprehensive description of the methods which are in industrial operation. It is written from a practical view-point and contains considerable detail on formulæ and procedures. The methods available include the chemical deposition of thin films of metals on the surface of non-metals to be followed by electro-plating, as well as metal spraying and cathode sputtering in a vacuum. Other publications on the subject, of a similar nature, have been written by Dickson[103] and Hepburn.[104]

Physical Metallurgy and Testing

Intercrystalline cracking and internal stresses.—In October the Institute of Metals held a symposium on internal stresses in metals and alloys, but relatively few of the numerous papers which it included dealt directly with copper alloys. That by Cook[105] on the relation of composition to stress-corrosion cracking in copper alloys, however, deserves special attention.

T'ing-Sui Ke[106] describes a research designed to examine thoroughly the mechanical behaviour of grain boundaries in metals in a quantitative manner. A simple torsional apparatus was devised for measuring certain anelastic effects at very low stress levels—namely, internal friction (damping) at low frequencies, variation of dynamic rigidity with temperature, creep under constant stress, and stress relaxation at constant strain. The apparatus is being used for examination of the viscous behaviour of the grain boundaries in metals. Evidence bearing on the causes of intercrystalline failure at elevated temperatures, especially under "creep" conditions, has been collected by Bleakney.[107] He states and criticises the existing theories of intercrystalline cracking and suggests that intercrystalline oxidation plays a far more important part in the process than is generally realised.

Oxidation and scaling.—The mechanism of oxidation and tarnishing is of a no less fundamental importance than that of intercrystalline cracking, and a paper by Ulick Evans[108] describes the different ways in which the thickening of oxide or sulphide films can occur. Normal migration of cations and electrons gives parabolic film growth, but mechanical breakdowns may lead to other equations.

In the course of exhaustive researches, using electron diffraction methods, on the nature of the oxide films formed on metals at elevated temperatures, Gulbransen and Hickman[109] have demonstrated that the oxidation of copper results in the formation of cuprous oxide alone, and found no evidence of chemical or structural transformations in the scale. While this is doubtless true of the thin films investigated by electron diffraction, it must be remembered that the black outer scale which forms on copper is essentially cupric oxide.

Rhines[110] who originally described subscale formation due to oxidation, largely on copper alloys, has reviewed this phenomenon in a recent publication. It is a diffusion process in which the more reactive component of an

alloy is oxidised by the inward diffusion of oxygen. The rate of advance of the front of oxidation is controlled by the relative diffusion rates of oxygen, and of the reacting element in the alloy. Precipitated oxides appear to influence the modes of recrystallisation and grain growth but attempts to show any hardening effect analogous to age hardening proved fruitless.

Metallography.—Apart from a careful investigation by Mack[111] of the structural changes which take place during the heat-treatment of eutectoid aluminium bronze, little of interest has appeared concerning the metallography of copper alloys. Mack and Shurman,[112] however, have described an etching reagent which darkens the delta constituent of aluminium bronze. This consists of the well-known mixture of ammonia and hydrogen peroxide which has been allowed to stand for from two to nine days in a loosely stoppered bottle. The concentration of the solution is said to be far less unimportant than the period for which it is stored; the time must be neither too long nor too short. No reason for the effect of storage could be adduced, and all attempts to hasten the maturing of the solution, such as by warming and aeration met with no success.

Scrap and Industrial Waste

Wise, Dodge and Bliss[113] have described the operations of the brass and copper industry which lead to wastes of various kinds, reviewing briefly figures for the amounts of sulphuric acid, sodium chromate and metallic zinc and copper consumed or lost in any one year. Data for the amounts of the main components in both the concentrated and dilute waste liquors from the pickling of brass are presented, and it is shown that, for one plant at least, approximately 90% of the pollution load was in the more dilute wastes and rinse waters. This rather startling conclusion, arising from the relatively huge volume of the rinse waters, should provide food for thought for all who are concerned with pickling plant. Several treatment processes for the recovery of the metallic content of waste liquors are outlined and illustrated by flow diagrams.

Recently the Non-Ferrous Scrap Metal Merchants' National Association[114] have drafted specifications for use in connection with the export and import of non-ferrous scrap metals. While the use of this standard classification is by no means compulsory, it is clearly a desirable advance that it should have been made. Full details, with code words, will be found in the reference mentioned.

A Century of British Progress

This account of progress in 1947 is brought sharply into perspective by a broader review, by Chaston,[115] of the contribution which Britain has made to progress in non-ferrous metallurgy during the century now ended. Much of the paper is devoted to a description of the work of eminent and pioneer metallurgists of this country, covering all branches of the science, but special mention is made of the fact that in the early years of last century about 75% of the world's total output of copper was smelted in South Wales, and that origination of the electrolytic refining process for copper can be attributed to Elkington of Birmingham. It might be added that earlier still another Englishman, Emerson, was the first to use metallic zinc instead of calamine ore in the manufacture of brass. The earliest freezing point diagram for any series of alloys was that determined by Roberts Austin in 1875, for the silver-copper system, and from that time onward copper and copper alloys have received much attention from workers in this country, largely under the auspices of the British Non-Ferrous Metals Research Association since its inception in 1920. The researches of D. Hanson and his collaborators on the effects of impurities in copper have become classical, while gas unsoundness in copper, bronzes and similar alloys are associated with the names of N. P. Allen, W. A. Baker and Pell-Walpole. Maurice Cook and his associates have made contributions of fundamental importance to a study of the cold working and recrystallisation of copper, and have provided a vast body of information on the mechanical properties of wrought copper-base alloys under various conditions of cold-work. The contribution of Britain to the metallurgy of copper and its alloys has clearly been by no means inconsiderable.

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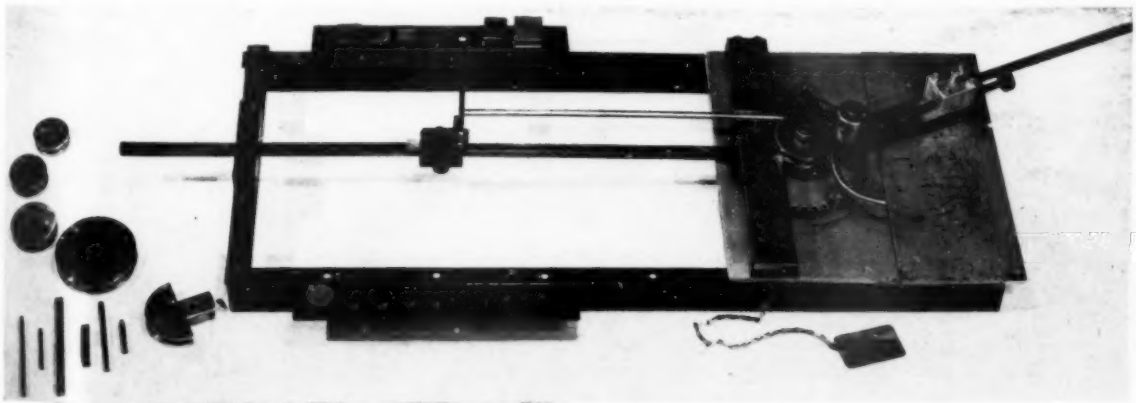
A New Tube, Bar and Rod Bending Machine

A NEW machine has recently been introduced by Tungum Sales Company Ltd. to provide a more accurate method of tube, bar and rod bend, known as the Branda repetition bending machine. Although following the normal method of tube bending, it incorporates several new features, particularly a system of accurate control on the length of the tube and the angularity of bend, which make it very versatile within its capacity. The machine is hand operated and may be used for manipulating tube, bar, rod and wire; tube can be bent cold and unloaded. There are two models; small and medium, the former for manipulating up to ½ in. outside diameter and the larger model up to 1½ in. outside diameter. Both are precisely the same in design and formers for unfilled or plug filled bending are supplied for either machine and are interchangeable.

The accompanying illustration shows the smaller model. It will be noted that a length gauge slides along the central bar which is drilled and numbered, so that, in conjunction with stops, accurate lengths can be fixed. A reaction block-plate is also drilled and numbered to take an appropriate reaction block. This reaction block holds the tube in position whilst the bending is being carried out. The choice of a suitable block depends on the radius of the bend being applied. Alternative blocks are supplied with the machine. The former too, which is the central pivot of the machine, varies in size according to the radius required. By operating the bending arm, the slipper block is caused to bear on the tube by the roller, which can be adjusted. Ten stops are arranged to give a 1° alteration in angularity.

When using the machine for tube bending, the tube to be formed should be cut to length and all burrs removed. The stops are then set and the tube fed between the former and reaction blocks to make contact with the gauge length arm. The roller is then screwed up in the normal way. The gauge-plate should be inserted between the roller and the slipper-block, to act as a feeler gauge. When the slipper bears firmly on the tube, the gauge plate should be withdrawn and the bending arm brought smoothly round to the angular stop. The sequence of operations ensures accurate reproduction.

This machine has already been successfully applied



The "Branda" repetition machine.

to the forming of gas, steam and hydraulic pipes; domestic water systems; brass and copper tube in general engineering applications; electrical conduit; and bars and rod for reinforced concrete and structural engineering. Thus, it has many and varied applications.

High Frequency Induction Heater

EDDY current losses and dielectric losses that are normally headaches to the radio engineer are being used on an increasing scale as methods of industrial heating. The former is used for the heat treatment of metals but, until the introduction of the Philips F.V. 100 generator, a large number of operations and processes that could benefit from this method have been outside the scope of the equipments manufactured because the power required has been beyond the capacity of existing valve driven plant.

The Philips F.V. 100 generator will produce up to 54 kw. *measured in the work*, (rated figure 50 kw.) and true skin heating as opposed to skin hardening can be achieved on areas of 7–10 sq. ins. This brings within the scope of the process a very large number of components which cannot be treated progressively and in which "single-shot" treatment with lower powers results in heating to such a depth that the mechanical properties of the material are impaired.

The range of work possible is as wide as that handled by the smaller generators now in use but the size of specimen may be larger or the number greater. For example, $\frac{3}{4}$ in. dia. steel bar hardened to ·030 in. at 2–2$\frac{1}{2}$ ft. per min. with 20 kw. can be hardened with 50 kw. at a speed approaching 8 ft. per min. or where 20 kw. will melt 10 lbs. of steel in 10 mins., 50 kw. will melt 25–30 lbs. in the same time.

The generator has been designed for flexibility, to cope with the variety of work it has to perform. In addition to the main frequency of 350 kc./s. R.F. current is available at 400 kc./s., 700 kc./s. and 1,000 kc./s., the frequency being selected by an off-load link system. The main purpose of the additional frequencies is to provide large steps in the output current so that widely different loads can be matched to the generator. By this means, the full power output of the generator is available in masses of metal whose volume varies as widely as from say $\frac{1}{10}$ cubic inch to a cubic foot.

One of the most prominent features of the generator is the on-load continuous variation of output current. The ratio is approximately 2 : 1, which gives a variation of 4 : 1 in the power in the work. Work coils do not have to be wound to very accurate limits to obtain the maximum output of the generator. Melting is made easier and quicker. In particular, it does away with a lot of tedious preparation in carrying out pre-production experimental work. This on-load variation is obtained by inserting a variable step-down R.F. transformer between the valve anode and tank of the Colpitts circuit. An advantage of this transformer is that it is almost loss-free.

The physical dimensions are less than those of some generators of 25 kw. rating. Servicing and maintenance have been carefully considered in arranging the general layout of the components, all of which are accessible from the two rear doors and the internal gangway. The standard model is suitable for main voltages of 380–440 v., 3-phase, 50 cycles. The maximum input is 93·5 kw. at a power factor of approximately 0·9.

The Philips high frequency induction heater, type F.V.100

"Wayne" Patent Portable Standard Type Self-Winding Electric Cable Reel

CONSIDERABLE interest attaches to the new "Wayne" patent portable standard type self-winding electric cable reel, which has the advantage of being easily attached to the wall by means of a fixing arm from a small pivot. In these circumstances the ceiling fixing flange is eliminated, being replaced by a neat handle securing boss which also secures the radial arm.

The "Wayne" patent portable standard type self-winding electric cable reel.

The object of this new design, which absorbs 25–30 ft. of 3-core cable, two conductors, and one earth, is to allow the use of one or two reels in a large workshop at low level instead of the other types of "Wayne" reel provided with a ceiling fixing flange at a high level. In this connection also a three-pin plug is supplied as an extra.

Increasing use of portable hand-operated electric tools in many industries makes the portable standard reel exceedingly attractive, since not only does it save damage to the cable but also provides a greatly added measure of safety in the use of portable tools in general. This new design constitutes an ingenious modification of the "Wayne" standard reel, developed by Mr. David Rushworth, Managing Director of Power House Components Ltd., the manufacturers of the reel, under the trade mark "P.H.C."

The principle of the reel is that of the spring roller blind, the cable being pulled out as required against the action of a spring which ensures it is kept taut, and on release springs back again, being something like a clock in a metal casing. This results in a great improvement in the working of electric cables of all kinds, including for example, reduced wear and tear and maintenance costs, while as indicated in the case of portable electrically operated tools there is much increased safety.

Type "P.V." Electrode

INCREASING attention has been paid in recent years to the X-ray examination of welds for many purposes. In particular, welded pressure vessels constructed to Lloyd's Class I requirements, or the APPI–ASMI code, which are subjected to very thorough inspection. Every inch of weld is examined radiographically and must be substantially free from porosity and slag inclusions. The choice of electrode for this class of work, it is claimed, has hitherto been limited to a few types of American origin.

Particular interest is taken, therefore, in the production by Murex Welding Processes Limited of their Type "P.V." Electrode, claimed to be the first all-British arc welding electrode designed specifically for pressure vessel welding. In addition to attaining a high standard of radiographic cleanliness, the weld deposits have excellent mechanical properties. The following are the average results obtained on a number of all-weld specimens made with No. 6 gauge and $\frac{1}{4}$ in. diameter electrodes.

Yield Point	24·2 tons per sq. in.
Ultimate Stress ..	29·9 tons per sq. in.
Elongation on 1½ in.	31·4%
Reduction of Area	50·6%

These all-weld specimens were machined out of U and Vee butt-welds in $\frac{3}{4}$ in. boiler quality steel, and tested after stress relieving at 600° C. Specimens from the same butt welds were subjected to Izod and bend tests, the results being as follows :—

Izod.—Notch at centre of top face of weld, 55 ft. lbs. ; Notch at junction of top face of weld, 41 ft. lbs.

Bends.—All specimens bent 180° over a 3T former.

The Type "P.V." Electrode is designed specially for ease of running in the downhand position in deep groove multi-run welds. It is suitable for both D.C. (60 volts O.C.) and A.C. (80–100 vols O.C.) supplies, but D.C. supply is recommended for superior radiographic results. It is available in various gauges to suit all plate thicknesses.

While primarily intended for pressure vessel welding, the electrode can advantageously be used in work which is to be vitreous enamelled, or for vacuum tight equipment. Technical information on current values, plate preparation, storage and welding techniques appertaining to this electrode can be obtained from the manufacturers, Murex Welding Processes Limited, Waltham Cross, Herts.

The Cleaning of Metals

Part III—Further Operations

By R. Groves

For many parts more than one cleaning operation is necessary and in this article operations are discussed for cleaning buffed and polished parts and for electroplating. Particular attention is directed to degreasing solvents, their advantages and drawbacks, and to cleaning by petroleum distillates.

PARTS that have been given a coating of heavy oil to prevent rusting in transit or storage, or have been painted or otherwise treated, can be cleaned in the same way as oil quenched parts, but if the oil used refuses to respond to this treatment, it may be necessary to divide the cleaning operation into two stages, a prior cleaning with an emulsifiable cleaner, either in a still tank or sprayed on, or with a degreasing solvent, followed by the alkaline solution as for quenching oils.

Cleaning Buffed and Polished Parts

Many parts that have been buffed or polished are required for electroplating, in which case a two-stage operation is again required. The first cleaning is essential as a means of eliminating the superficial matter, which may be grease, soot, oil, paints, abrasive materials, lithopone, emery, lime, crocus, tripoli, rouge, metallic particles, etc., according to the processes to which the parts have been subjected. As will be imagined, these do not all present by any means the same problem, and in many instances special cleaning agents and compounds may be required. Nevertheless, for a number of jobs it is possible to employ certain standard materials and processes.

Whatever the metal of which a buffed or polished part is made, it can be precleaned by means of a good quality emulsifiable cleaner sufficiently to provide a surface that can be painted, lacquered or finished in

any other manner with the exception of electroplating, for which special treatment is necessary.

Cleaning for Electro-plating, etc.

Electroplating demands a surface that has no microscopic superficial cracks, has no oily or greasy area that prevents the electrolytic solution from "wetting" it, and has no minute irregularities. Such a surface is also essential for vitreous enamelling. Although electrolytic cleaning is advocated by many firms because it is a single operation, it is not always effective, and in the main the double operation of precleaning to remove the rough dirt, and electrolytic cleaning with a comparatively clean solution afterwards, is preferable. This not only makes it easier to choose the correct finishing solution, but also means that the finish cleaning is confined only to such foreign matter as remains in the "pores" or crevices, too small to be seen except with the microscope and any traces of oil or grease on the surface that might prevent "wetting."

Types of Cleaner for Finishing

An alkaline solution of specially high quality is advisable for the finish cleaning of steel parts, wherever dipping is adopted or washing machines are used, because it is swift in action, gives a perfect wetting of the surfaces, and penetrates more effectively into the "pores" of the metallic surface than the majority of other alkaline solutions, while when used on parts of steel, it tends to give a brighter finish. It is also suitable for use in electrolytic tanks, when this form of cleaning is used, and if necessary can also be used for those parts that do not require to be pre-cleaned for plating or enamelling.

For parts of non-ferrous metals or those other than steel, it is necessary to use specially compounded cleaners whose composition is determined by the kind of metal. For example, a different solution will be required for soft die castings than for the brasses; and a different solution again for parts in which combinations of metals occur. Again, there will be differences in the cleaners required according to whether the part is dipped, cleaned mechanically, or electrolytically cleaned.

Current practice is to employ anodic cleaning for the final operation, and the solution chosen for this purpose should be capable of carrying adequate current to produce the high current densities required, while in addition it must contain no ingredient liable to set up anodic corrosion. The cleaners used must also be extremely effective in penetration, wetting and dispersal. They should have the effect of lowering the surface tension of the solution, which means that the parts can be drained much more quickly and with a minimum of drag-out loss. Rinsing accordingly can be speeded up and becomes more effective, so that the plating electrolytes, etc., used later are less liable to be contaminated by the residues of the cleaning solution and are not impeded in their work by surface films forming on them.

Details of Operation

The electro-cleaning agents operate instantaneously, and for steel, brass, and die cast parts should be employed at concentrations of 12, 6 and 4 oz. per gallon, respectively. Steel should be cleaned at 100° C., brass at 70–80° C. and die cast parts at 80–90° C. The current density required will be of the order of 50 amps. per sq. ft. The cathodes should, for preference, be of perforated steel nickel-plated.

Degreasing Solvents

Chlorinated solvents for the cleaning of metals have been in use for well over twenty years, and have been greatly improved. The principle of these cleaning agents is as follows. The part to be degreased is, while still in the cold state, hung up in a vapour given off by the solvent when boiled, and this vapour is both at a high temperature and comparatively heavy. As the parts are at a lower temperature than the solvent, the vapour condenses on their surfaces, and the result is a liquid which causes accretions of oil and grease to dissolve, on the assumption that their composition is such that they are soluble in the solvent liquid. The eliminated materials trickle down with the liquid and fall back into the boiling solvent in its container. It will be obvious that there must be instances in which the oil or grease is not soluble in the liquid, but this is not the only drawback. The rate at which the condensate flows over the parts is too slow to remove every trace of such materials as lime, paints, metallic particles, and solid dirt generally, which in any event are not dissolved by it. Consequently, it is often necessary to complete the cleaning by hand-rubbing, by additional cleaning processes, or by degreasing in two or more stages.

Improved Solvent Degreasing

In any event, if the part has to be electro-plated or vitreous enamelled after degreasing, the solvent treatment will not be enough, because it does not leave a *chemically* as distinct from a physically clean surface. It is also by no means easy to obtain even physically clean surfaces without a later hand wiping or spraying operation.

To overcome these difficulties, it is common practice to-day to carry out degreasing in two or more stages. The first is a steeping of the part in, or spraying with, the solvent, or alternatively, its immersion in a tank of solvent, which is mechanically agitated. In some instances the work is agitated in the solution. This certainly helps to eliminate a proportion of the solid dirt, but one such immersion and agitation is rarely sufficient, and modern degreasing machines operate in the following manner. The part is first soaked in the solvent, given a second soaking in a different tank, then sprayed with the solvent. The next stage is to cool the parts, either by steeping them in a tank of the cold solvent, or by placing them in a refrigerated zone. Lastly, the normal vapour degreasing is carried out as a final treatment, for rinsing and drying.

It will be seen that spraying of the solvent is an essential feature of this improved process, but as earlier stated, spraying of the degreasing solvents is by no means easy, and necessitates high pressure if the more inaccessible areas are to be attained. Even then, the parts are not given the chemically clean surface required for certain work, and a final alkaline cleaning becomes necessary.

Advantages and Drawbacks of Solvents

However, the degreasing solvent has certain advantages. It is valuable as a means of removing oil and grease from small metal parts having rolled edges, recesses, pockets, seams, etc., from which the draining of an aqueous solution, or its rinsing and drying off, would be difficult. It is also useful for cleaning off lubricants or rust-preventives as long as these can be dissolved in the solvent. In addition, it is specially

advantageous for those parts of non-ferrous metals and alloys that are liable to tarnish when treated with water solutions and rinses.

The drawbacks, over and above those already enumerated, include the liability of ferrous metals to rust quickly after the treatment, so that they must be given a coating of rust-proofing material, paint or lacquer immediately degreasing is completed. Aluminium also presents difficulties, because parts of this metal and its alloys that have been ground or polished usually carry on their surface a number of extremely small particles of aluminium. These have a catalytic action and disintegrate the trichlorethylene solvent, resulting in the formation of hydrochloric acid. This leads to corrosion of both the parts and the bath.

Solvent degreasing with trichlorethylene is also more expensive than either alkaline or emulsifiable cleaning processes, not only because the cleaning material itself is more expensive, but also because the water for the condensing coils has to be cooled, which is an item of cost to be considered.

Against this, however, must be set the fact that it costs more to heat an alkaline solution than to heat the solvent, and in this respect the emulsifiable cleaner is probably the least costly of all.

Loss of Solvent

In order to minimise solvent loss it is essential to use a well-designed, closed type of cleaning machine, but even this will not prevent loss, which is an important point. It is reported by one industrial firm that a degreasing solvent costs from twice to four times as much as an emulsifiable cleaning material per sq. ft. of surface cleaner, and that on this basis the alkaline solution is the most economical of the three.

Although water is required for all three types of cleaning process, it is often the case that more is needed for vapour degreasing than for either of the other two processes, even though it is needed solely for condensation. Moreover, even a well-designed and built degreasing machine rarely lasts more than a few years, as compared with the machines used in the other processes, which have often a life of ten or twenty years.

Perchlorethylene

Because of these disadvantages, the latest practice is to use perchlorethylene as a solvent degreaser, and this has certainly effected notable improvements. No water is needed for condensation, by reason of the considerably higher boiling point. Thus, cooling systems can be dispensed with, thus reducing cost, and in addition can be made portable, because no water piping is required. As the solvent condenses at a temperature much higher than 100° C., no catalytic action takes place, as outlined earlier, because no water can remain in a system of this type.

Perchlorethylene vapour is heavier than that of trichlorethylene, roughly in the proportion of 3 to 2, and in consequence less of the solvent is lost in the form of vapour. The greater weight of the solvent makes it more suitable for use as a spraying medium, so that effective spray systems are readily designed for pre-cleaning in a machine. Actually, the solvent can, because of its greater weight in both liquid and vapour form, even be sprayed in uncovered tanks in certain cleaning process. It is less poisonous and its use presents less risk.

There is also a form of " inhibited " perchlorethylene, commonly known as tetrachlorethylene.

Cleaning by Petroleum Distillates

Cleaning with petroleum products differs from the forms of metal cleaning hitherto described in that it is not designed to produce a chemically or even a physically clean surface in readiness for other processes such as plating, enamelling, etc. It is, however, an important form of industrial metal cleaning. It is intended to remove from metallic parts such fairly easily handled substances as dust, swarf, shop dirt, etc., in advance of assembly, or, on occasion, after assembly. It is also capable of cleaning off slight films of grease, oil or protective coatings. Because it does not corrode, tarnish or rust the parts, it can be used for many cleaning operations that no other material would achieve, e.g., the cleaning of delicate mechanisms or complicated assemblies that would be stained or corroded by alkaline solutions, and could not anyhow be water-rinsed afterwards. The same applies to the emulsifiable cleaners, which have also to be rinsed off with water. The solvent degreasers could not be used, because rusting follows too quickly on their employment.

The petroleum distillate cleaners are, as will be realised, highly voltatile, and have flash points so low that there is inevitably some danger in their use. Great care must, therefore, be exercised. They ought never to be employed in open tanks, and the machines used should be specially designed and constructed for the purpose, being provided with proper protective appliances and means for extinction of any fire caused.

In the next part a few more details of these cleaners will be given, and the emulsifiable cleaning materials discussed.

Wild-Barfield Form Indian Company

Personnel Trained by Parent Company

WILD-BARFIELD ELECTRIC FURNACES LTD., has formed a new Company known under the name of Indian Wild-Barfield Co., Ltd., with offices at Fort Chambers, 6-10, Dean Lane, Hammam Street, Fort, Bombay. The local management is under the control of Mr. R. A. P. Miara, who recently returned to India after a prolonged stay in England, during which he gained a thorough knowledge of the design construction and practical uses of Wild-Barfield furnaces.

The Company has been formed to cover sales and service of standard Wild-Barfield furnaces and equipment in the dominions of India and Pakistan and adjoining countries, and to carry out assembly and other constructional work as occasions demand. In addition, the Company has been appointed sole distributor and agent for the productions of Messrs. G. W. B. Electric Furnaces Ltd., including the larger mechanically operated heat-treatment equipment and Eternite case-hardening compounds, etc.

For many years, Wild-Barfield interests in the Indian field have been in the hands of Messrs. Alfred Herbert (India) Ltd., and the formation of the Indian Wild-Barfield Co., Ltd., supplements rather than supplants the long co-operation. In fact, the new Company may be reached through any of Messrs. Alfred Herbert's branches at Bombay, Calcutta, Kuala Lampur, Lahore and Madras.

Modern Methods of Gas Analysis*

Part VII—The Graham-Lawrence Apparatus and the McLuckie Gas Detector

By W. D. Vint

The increased use of blast furnace gas in steelworks has given rise to the problem of protection for those engaged on furnaces in which it is used. For this purpose an apparatus capable of detecting small amounts of CO in the atmosphere is necessary, such an apparatus is discussed. Reference is also made to an instrument used for the rapid estimation of inflammable gases in the atmosphere.

THE modern trend towards the increased utilisation of waste blast-furnace gas with its high carbon monoxide content in large scale complex heating furnaces has given rise to the problem of protection for those engaged in close proximity to the furnaces. By virtue of its 25/30% CO, blast-furnace gas is about five times more lethal than town's gas and in addition, the calorific value being so much lower, relatively large volumes of blast-furnace gas must be burnt to produce an equivalent of heat.

The detection and estimation of small amounts of CO in the atmosphere close to the furnaces is thus becoming increasingly important. A recent categorised report on annual industrial accidents showed a considerable percentage due to carbon monoxide poisoning, and it should, therefore, be stressed that any works, no matter how small, in which gases containing appreciable amounts of CO are utilised, should provide themselves with an apparatus capable of detecting minute amounts of CO in the atmosphere.

There are several types of quick-acting pseudo fool-proof instruments upon the market whose makers have endeavoured to give the impression that CO detection is an extremely simple process and quite capably carried out by any person able to read the "book of words." The only accurate and reliable test for CO involves a laboratory operation and is best done on the Graham-Lawrence apparatus which is sound in principle and operation.

Principle of the Graham-Lawrence

The apparatus depends for its successful working upon the fact that when carbon monoxide is passed over heated iodine pentoxide an equivalent amount of iodine is released.

$$5CO + I_2O_5 = I_2 + 5CO_2$$

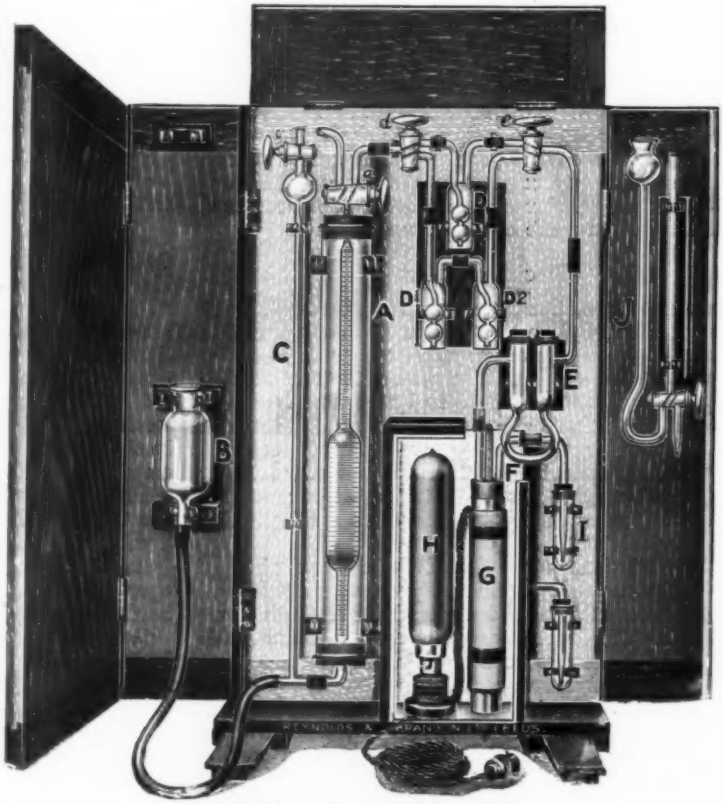
*Continued from October 1947 issue, page 334.

The sample of air containing the unknown quantity of CO is measured by volume at atmospheric pressure and temperature, passed through a suitable train of absorbents to rid it of unwanted gases and moisture and finally through an iodine pentoxide tube electrically heated to a temperature of 120° C.

The equivalent iodine released distils over into a test tube containing 10% KI solution, is absorbed, and the solution titrated with a standard sodium thiosulphate solution using starch as indicator. The volume of CO can thus be calculated and after due correction to N.T.P. on the original air volume the percentage of CO can be calculated.

Description of the Apparatus

The gas is measured into the 200 cc. water jacketed graduated gas burette (A) and the temperature of the water (and hence the gas) is indicated by a thermometer which can be inserted at the top of the burette (the thermometer is not actually shown in the pictorial view of the apparatus). Mercury is used as the



The Graham-Lawrence Apparatus

confining liquid, and the reservoir shown at B is about 500 ccs. capacity. The air or gas sample is introduced at the side limb of the tap "a" by the usual method of water or brine displacement. Two small scrubbing vessels at D1 and D2 provide a means of removing unsaturated hydrocarbons which, if present, seriously interfere with the iodine reaction. The scrubbing vessels D and D1 each contain a few ccs. of strong KOH solution, whilst D2 contains bromine water. If it is known that the sample of gas contains no hydrocarbons the vessels D1 and D2 may be bypassed by operation of taps "d" and "e" in which case the sample then passes through D only. Unwanted CO_2 is removed in either case and the sample then passes through the U tube E. Three separate reagents separated by dried glass wool are packed into this tube and the gas passes through them in the following order: (1) Soda asbestos which removes the last traces of unwanted CO'_2 and incidentally a certain amount of moisture. (2) Activated charcoal which further rids the gas of unwanted hydrocarbons. (3) Phosphorous pentoxide which removes the last traces of moisture. The latter operation is extremely important as it is essential that the gas entering the iodine pentoxide tube is perfectly dry.

The iodine pentoxide tube itself in the shape of a U is enclosed in a larger tube G which is electrically heated from the mains. H is a resistance of such value that the temperature of the tube (as indicated by the thermometer) is kept constant at 120° C. After the Iodine Pentoxide tube the gas is bubbled through 10 ccs. of 10% KI solution contained by the test tube at I

Procedure for Analysis

A blank should first be made by passing 200 ccs. of air through the apparatus and estimating the equivalent of CO released from the iodine pentoxide tube. This blank estimation is carried out in exactly the same way as the subsequent analysis is made.

The gas is measured into the 200 ccs. water jacketed gas burette (A). If it is known that a fairly high percentage of CO is present in the sample a smaller portion is taken for analysis and it should be arranged that in the final titration no more iodine is released than would be equivalent to about 3 ccs. of sodium thiosulphate solution. If a greater portion of gas is taken than this, solid crystals of iodine will condense on the outlet limb of the U tube and necessitate extensive sweeping with air.

The gas sample is measured in the usual manner and a note taken of the jacket temperature and prevailing barometric pressure. The gas volume may now be converted to volume at N.T.P. Potassium iodide solution is now added to the test tube W and connection made to the U tube. The temperature of the iodine pentoxide tube is noted and should be 120° C. The reservoir B is now clipped to the highest bracket at the side of the water jacket and the tap "a" very cautiously opened. A steady trickle of gas the, speed of which is indicated by the bubbling in the test tube is allowed to pass through the apparatus. The best speed is about 20 ccs. per minute.

When the mercury reaches the top of the gas burette, the tap "a" is reversed, the reservoir lowered and a burette full of fresh air taken in. The reservoir is again clipped to the top bracket and tap "a" partially opened. Air is now passed through the apparatus at approximately the same speed as before to sweep out the remains of the gas enclosed by the U tube and scrubbing vessels. The presence of CO in the gas is at once indicated by a yellow colour appearing in the KI solution. When the 200 ccs. of fresh air has been passed through the apparatus, the test tube is detached and the outlet of the iodine pentoxide tube sealed. A solution of sodium thiosulphate made up as follows is used for titration.

Stock solution—5·541 grams sodium thiosulphate dissolved in water and made up to 250 ccs. Two ccs. of the above solution are now taken and diluted to 100 ccs. The weaker solution which must be made fresh each day (the stock solution will last a week) is now poured into the special 1 cc. burette provided with the apparatus and the contents of the test tube titrated, using starch as indicator.

A calculation typical of this type of apparatus is as follows:—

Volume of air taken for analysis .. .. .. 193·6 ccs.
Barometric pressure .. 755·5 mms.
Water jacket temperature .. .. 21·0° C.

Volume of air corrected to N.T.P. (refer to Methods of Air Analysis by Haldane and Graham, page 55)—

$= 193{\cdot}6 \times 90{\cdot}01$
$= 174{\cdot}3$ ccs.

Titration: 2·34 ccs. of sodium thiosulphate used.
Blank: 0·07 ccs. of sodium thiosulphate used.

1·00 cc. of sodium thiosulphate solution is equivalent to 0·1 cc. CO at N.T.P.

Therefore, CO in sample $= \dfrac{2{\cdot}27 \times 100}{174{\cdot}3 \times 10}$
$= 0{\cdot}130\%$

Sample tube used for checking Graham-Lawrence apparatus.

Precautions to be Observed with the Apparatus

1. The Iodine Pentoxide tube must never be allowed to become moist. It is a good plan to leave the heater switched on even when the apparatus is not in use.

2. Care should be taken that the KI solution is not drawn up into the Iodine Pentoxide tube.

3. The phosphorous pentoxide drying tube must be changed fairly frequently if the apparatus is in constant use.

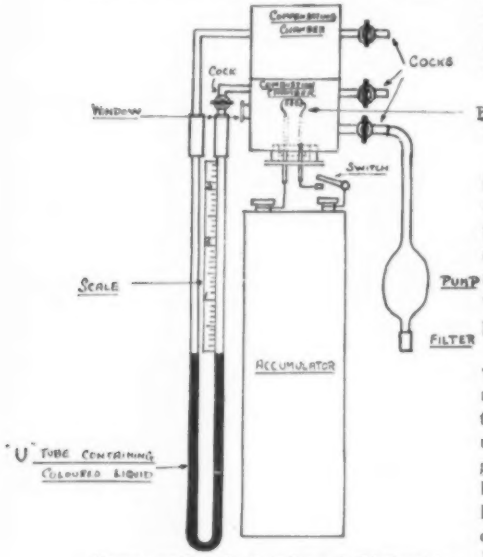
Method of Checking Graham-Lawrence

The apparatus may be tested quite simply, and it is advisable that checks are made from time to time.

The sampling tube as shown in the sketch (the volume of which is known accurately) is filled with town's gas and after clearing the outer capillaries, the gas is admitted to the gas burette. Air is also added to dilute the sample and bring the volume up to about the 200 ccs. mark. The gaseous mixture is then passed through in the usual manner and the CO estimated. The town's gas from which the sample was taken is also analysed for CO (on the Orsat apparatus preferably) and from this percentage figure the volume of CO passed through the Graham-Lawrence can be calculated. This latter figure should check up with the CO as found by the Graham-Lawrence.

The McLuckie Gas Detector

This instrument is used for the rapid estimation of inflammable gases in the atmosphere and is indispensable for oil fields and refineries, or wherever inflammable gases are utilised. The apparatus is simple and speedy to use and tests at the rate of ten an hour can be carried out on it.



Constructional details of McLuckie gas detector.

The principle upon which the McLuckie depends is the well-known reaction produced when a combustible gas is passed over a hot wire. In the case of methane, for example, the reaction is as follows :—

$$CH_4 + 2O_2 = CO_2 + 2H_2O$$

The methane combines with twice its volume of oxygen and produces CO_2 and steam (H_2O). The steam produced is twice the volume of methane burned, consequently, when the steam condenses, a reduction in volume or a reduction in pressure is produced.

The apparatus consists of a metal vessel divided into two equal compartments of which the lower comprises the combustion chamber and the upper a compensating chamber. The gas in the combustion chamber is burnt by means of a platinum wire, heated from an accumulator, and the difference in pressure in the two chambers accruing from the combustion is measured by the U tube.

The multiway cock is used to admit air into the combustion chamber, to close all passages during combustion and to make connection between the two chambers and the U tube. The gas under test is forced into the combustion chamber by opening all the passages to atmosphere and by operating the small hand blower. By turning the handle to position 2 the passages are all closed and after two minutes have elapsed (which is sufficient time to allow an equalisation of temperature in the two chambers) the handle is turned back to No. 1 position. The pressure in each chamber now becomes atmospheric, the handle is again turned to No. 2 position and the electric current switched on for two minutes. The combustion may be observed through a small window fitted to the combustion chamber. After a further two minutes have elapsed the handle is turned to position 3 and the difference in pressure resulting from the condensation of the moisture formed by the burning methane may be read on the scale at the back of the U tube.

Philips Electrical Industries Ltd.

THIS Private Company has been formed as a Holding Company with a capital of £2,500,000 to acquire and hold certain shares of the Philips Group of Companies in Great Britain. It is purely internal reorganisation and regrouping, and has no external implications.

Mr T. W. Melling, M.B.E., Retires

MR. THOMAS WILLIAM MELLING, M.B.E., retired from the position of works manager of the East Hecla and Hecla Works of Hadfields Limited on December 31, 1947, after nearly 50 years' service with the Company. Although born in Sheffield, he spent many of his early years in Russia where his father was manager of works laid down by Charles Cammell and Co. at Kolpino near St. Petersburg, as it was then known, for the manufacture of duplex armour plate. Returning to this country when he was 14 he completed his education in Sheffield and subsequently joined T. W. Ward as a learner in the engineering shops, later being transferred to Charles Cammell and Co. He completed his apprenticeship with a period at the River Don Works of Vickers Ltd.

Mr. Melling became associated with Hadfields Ltd. in July 1898, serving for 12 years in the machine shops under the direction of the late Mr. Frank Ellison. By 1912 he had risen to the position of production manager, in which position he carried much responsibility during the 1914–1918 war, particularly in connection with armour piercing shells; for his outstanding work he received the M.B.E. award. In 1930 he was appointed assistant works manager, a local director in 1940 and also works manager of Hecla Works. His valuable services were further recognised in 1945 when he was appointed works manager of both East Hecla and Hecla Works.

Tom Melling, as he was familiarly known among his friends, possesses high qualities of leadership as well as outstanding ability in the machinist craft; and it is not surprising that all sections of the works, from the workmen to the directors, have presented him with many tokens of their esteem. There can be no doubt of the good wishes of all who have been associated with him that he should enjoy many years of retirement.

Tom Makemson Honoured

A SPECIAL meeting of the Institute of British Foundrymen to be held at the Waldorf Hotel, London, W.C.2, on January 16, will mark the twenty-first anniversary of the appointment of Mr. Makemson as secretary. Elected to membership of this Institute in 1917, he became honorary secretary of the Lancashire branch in 1923 and was appointed secretary of the Institute in 1926. In the latter year the International Committee of Foundrymen Technical Associations was formed and Mr. Makemson was appointed secretary, a position he has held since. He is also secretary of the International Committee on Testing Cast Iron, and since 1927 has been secretary of the Manchester Association of Engineers.

Mr. Makemson was the recipient of the 1947 award of the Institute of British Foundrymen's "E. J. Fox" Gold Medal in recognition of his exceptional services to the foundry industry as secretary of the Institute for 21 years and for his outstanding work during the period 1940-1946 as Joint Director and later Director of Iron Castings in the Iron and Steel Control of the Ministry of Supply.

Analysis of Copper and Nickel Slags and Mattes

By J. Kinnunen
Outokumpu Oy, Pori, Finland

Simple and rapid analyses of copper and nickel slags and mattes, with reasonable accuracy, are necessary in the copper and nickel industry and methods are described which have been found to give satisfactory results.

THE smelting of copper and nickel ores to mattes requires the frequent analytical control of slags and mattes. Therefore, and also for economical reasons, it is important that the different analytical determinations are as simple and rapid as possible, with sufficient accuracy. In literature the available methods are dispersed and, in fact, do not frequently concern copper and nickel slags and mattes. Under these circumstances, standard methods in the copper and nickel industry do not exist as is the case in iron and steel industry.

Analysis of Slags

Decomposition of sample.—Some, especially the chilled slags, are easy to grind finely and to decompose by HCl and HNO_3 without resort to fusion. The decomposition is, however, nearly always easily accomplished by adding some HF. The interference of fluorides is prevented by heating the solution with H_2SO_4 to fumes. This treatment is rapidly performed by shaking an Erlenmeyer flask with narrow mouth over a free flame by means of a pair of tongs. When transparent quartz flasks are used the silica is also removed without introducing additional elements into the solution, the flasks being but slightly attacked by HF. When a complete analysis is required, a great initial weight is taken and from its solution in a volumetric flask an aliquot part for each determination is pipetted. For quick assays, however, the use of small separate assay portions is frequently preferred.

In some rare cases, where the decomposition with acids and HF is incomplete or the use of HF or H_2SO_4 must be avoided for some reason, it must be resorted to fusion. In these cases the slag is fused with Na_2O_2 in an iron crucible, which is then chilled in compressed air. The melt is loosened by tapping with a hammer on the bottom of the covered crucible. This fusion method may also be used for separations. When the melt is leached with cold water and filtered with suction, the precipitate consists of Cu-, Fe-, Ni-, Co-, Ca- and Mg-hydroxides, SiO_2, Al, Zn, S, alkali metals and some Cu being in the filtrate. In some cases the use of nickel or "alsint" crucible and a mixture of Na_2CO_3 and Na_2O_2 is profitable.

SILICA.—Nearly all methods for determination of silica are based on its precipitation by means of acids. In details, however, there are so great differences that a suitable method, good arrangements, and modern equipments (aperiodic balance, oxygen furnace) shorten considerably the time for analysis. A rapid method to precipitate the silica is to heat the solution with H_2SO_4 to fumes. If a greater accuracy is preferred to the rapidity, HCl is used and the purity is determined by means of HF and H_2SO_4 as usual. The use of perchloric acid is also time-saving but dangerous. Other methods as gelatine method are hardly quicker and for the present of secondary importance.

Procedure.—1 g. of the slag is fused with Na_2O_2 and the melt dissolved in a 750-ml. Erlenmeyer flask with a little amount of water. The crucible is rinsed with some HCl into the Erlenmeyer and then 40 ml. of H_2SO_4 1:84 is cautiously added under the tap. The flask is heated over a free flame until fumes of H_2SO_4 are evolved or salts are beginning to separate. The flask is carefully cooled and 80 ml. of HCl 1:19 is added and boiled for a moment to dissolve all cinder. The solution is diluted with hot water to about 400 ml. and filtered with suction on twofold paper and pulp in a Buchner funnel. The adhering silica in the flask is carefully transferred with hot HCl 1 : 10 and water by means of rubber policeman into the filter, which is then ignited in a flat platinum crucible in a current of oxygen into SiO_2. If the slag decomposes completely with acids, the fusion may be omitted. The silica is then precipitated immediately after the decomposition with aqua regia by heating the solution with some H_2SO_4 above a free flame to fumes. The cooled residue is dissolved with water and some HCl and the silica is determined as above.

LEAD.—If lead is present it is precipitated as sulphate, free from silica, by decomposition of the slag by acids as described above. The precipitate is dissolved either by NH_4-acetate and lead is determined with molybdate method or, in presence of large amounts of calcium, it is dissolved by HNO_3 and lead is determined electrolytically as peroxide. For the determination of other components lead is easily removed as sulphate in the course of analysis. Frequently it does not interfere when present as insoluble sulphate, and thus the removing may be omitted.

COPPER.—There are many gravimetric, electrolytic, volumetric and colorimetric methods for the determination of copper. Owing to the homogeneous material and low percentage in slags a photometric method is suitable, Quandel[1] determines copper in steel, and Ringbom,[2] in ores by means of rubeanic acid. They use solutions buffered by acetate or tartrate mixture. The interference of iron is made harmless by NaF, or the iron tartrate colour is compensated. Other elements present in slags do not interfere. Also formiate and citrate mixture with or without NaF may be used.

Procedure.—0·1 g. of the finely ground slag is moistened with water in a 100-ml. silica Erlenmeyer flask and

1 Quandel, K., *Arch. Eisenhütten w.*, 14 (1940/41), p. 601.
2 Ringbom, A., *Metall. und Erz* 15/16, 1943, p. 228.

treated with aqua regia. When the reaction is over some HF is added to complete the decomposition. Then 5 ml. of H_2SO_4 1 : 1 is added and the solution heated over a free flame to fumes. The residue is dissolved with hot water, boiled and neutralised with NaOH. The slightly acid solution is transferred into a 200-ml. volumetric flask, 2 ml. of 1% gum arabic, 20 ml. of buffer mixture pH = 3·8 (50 ml. glacial acetic acid and 16·3 g. sodium acetate in 500 ml.), 10 ml. of 2·5% NaF and 4 ml. of 0·1% rubeanic acid in alcohol are added, diluted to mark, mixed and measured after 5 minutes photometrically with red filters. When necessary the solution must be filtered before measuring. When a greater accuracy is required, 2 g. of the slag is decomposed as above, and copper is separated by H_2S or $Na_2S_2O_3$ and determined electrolytically in the usual manner.

Iron.—The determination of large amounts of iron is based in technical analysis nearly always on titration either with $KMnO_4$ or $TiCl_3$. The interference of copper is easily eliminated by H_2S or ammonia.

Ferric Iron The determination of ferric iron is of interest, as it measures the amount of magnetite present in the slag.

Procedure.—1 g. of the finely ground slag is decomposed with 20 ml. of H_2SO_4 1 : 4, and some HF in a 500-ml. silica Erlenmeyer flask under CO_2-atmosphere. Fifteen ml. of HCl 1·19 is added to complete the decomposition. When the reaction is over, the solution is diluted with oxygen-free water, 2–3 g. of boric acid and 5 ml. of 10% KCNS are added and Fe + Cu is titrated with $TiCl_3$.

Alumina.—The classical methods for the determination of alumina in slags are tedious and inaccurate. The introduction of mercury cathode into analysis offers, however, an excellent means easily to remove most of the interfering elements. The use of oxine enables the volumetric determination of aluminium. Besides, it is also possible to recipitate aluminium with oxine in presence of many other elements according to Hexko,[3] Piggot,[4] and Reutel.[5]

Procedure.—0·5 g. of the slag is decomposed as above. The solution is neutralised by ammonia; 1 ml. of H_2SO_4 1 : 1 in excess is added. The solution is transferred into a mercury cell, diluted to 200 ml. and electrolyzed with 3–4 ampere. When the solution is decolorized, mercury is removed and the solution filtered and washed with hot water. Aluminium is then precipitated with ammonia and ignited at 1,200° C. to Al_2O_3 as usual, or precipitated with oxine and titrated as below. Aluminium may also be first precipitated together with iron as hydroxide by ammonia, avoiding the excess. The precipitate is dissolved with HCl, 8 g. of tartaric is added, the solution diluted to 100 ml. and made ammoniacal. To the boiling solution Na_2SO_3 is added until the colour turns lighter, and then KCN until the solution is yellow, and 3–4 g. in excess. Aluminium is then precipitated with 5% oxine in alcohol. When the precipitate has settled, the solution is filtered and washed with warm water and at the end with cold water. The precipitate may be ignited with oxalic acid to Al_2O_3 or dissolved wit'ı 50 ml. of HCl and alcohol 1 : 3. The solution is diluted to 100 ml., 0·5 n $KBrO_3$-KBr-solution is added by using methyl red as indicator, until the solution changes to yellow and about 2 ml. in excess; 1–2 g. KJ is added and the solution titrated with n/10 $Na_2S_2O_3$ starch as indicator.

Zinc.—The lack of a rapid, accurate, and universally applicable method for the determination of zinc is generally acknowledged. The interfering elements, such as copper and iron, must be first removed and zinc is the precipitated in a slightly acid sulphate solution by H_2S and ignited to oxide. A double precipitation and the retreating of filtrate is often recommended. The iodometric K_3 [Fe(CN)$_6$]-method as described by Land and Reifer[6] is, however, suitable for the slags. The method fails in presence of nickel and cobalt, but then zinc may be first precipitated by oxine, as described by Berg.[7]

Procedure.—0·5 g. of the slag is decomposed as above; 3 g. of tartaric acid is added and the solution neutralised with 2 n NaOH 15–20 ml. in excess. Zinc, copper and magnesium are precipitated with 2% oxine in alcohol. The solution is heated to 60° C., cooled, filtered, washed with cold slightly alkaline 1% sodium tartrate and at the end with cold water. The precipitate is decomposed with HNO_3 and heated with 5 ml. of H_2SO_4 1 : 1 to fumes. The residue is diluted to 75 ml. and ammonia is added, until a little amount of iron is precipitated. To the cooled solution KHF is gradually added, until the solution changes colour and then a little in excess. KJ is added and J titrated with n/10 $Na_2S_2O_4$ starch as indicator. At the end some NH_4CNS is added. For the determination of zinc K_3FE (CN)$_6$-solution (66 g./l) is then added from burette in portions of 2 ml., and J is titrated with n/10 $Na_2S_2O_3$.

Nickel.—Nickel in slag is easily separated from interfering elements with dimethylglyoxime and determined gravimetrically or electrolytically or colorimetrically according to Bauer and Eisen.[8] For small amounts of nickel the use of solid reagent is preferred.

Procedure.—0·2 g. of the slag is decomposed as above; 0·6 g. of tartaric acid is added and the solution is made slightly ammoniacal. Nickel is precipitated in a boiling solution with 100–150 mg. of solid dimethyloxime. The cold solution is filtered and washed with hot water. The precipitate is decomposed with HNO_3 and heated with some H_2SO_4 to fumes. The residue is diluted with water, 5 ml. of bromine water; 5 ml. of ammonia 1 : 1 and 3 ml. of 1% dimethylglyoxime in alcohol are added. The solution is diluted to 100 ml. and measured photometrically with blue filters.

For greater accuracy 2·5g. of the slag is decomposed and precipitated as above and nickel is determined electrolytically or gravimetrically as usual.

Cobalt.—The accurate determination of cobalt in the slags is tedious and difficult. The interfering element must first be removed, iron in a hydrochloride acid solution by extraction with ether, copper and zinc by H_2S. Cobalt is then estimated together with nickel as metal, reduced by electrolysis from an ammoniacal solution. In presence of large amounts of aluminium the hydroxides are contaminated by cobalt and so the results are low. The use of α-nitroso- β-napthol simplifies the determination considerably, but not sufficiently. The reaction of Vogel makes it, however, possible to determine cobalt colorimetrically as described by Tomula.[9] The method, modified by Ringbom,[10] for ores is also applicable for slags.

3 Hexko, T., *Chem. Z.*, **58**, 1032, 1934.
4 Piggot, E. C., *J. Soc. chem. Ind.*, **58**, 139, 1939.
5 Reutel, C., *Metall. und Erz.*, 1941, **8**, p. 170.

6 Lang, R., and Reifer, J., *Z.f. anal. Chem.*, **93** (1933) S. 161.
7 Berg, R., *Die analytische Verwendung von Oxychinolin S.29 u. 42.*
8 Bauer, Eisen, *Metall. und Erz* **21**, 1940, p. 433.

Procedure.—0·5 g. of the slag is decomposed as above. To the slightly acid boiling solution 5 ml. of 10% hydroxylaminehydrochloride and some 20% NH_4CNS are added, boiled and cooled. The solution is diluted in a 100-ml. volumetric flask to mark, mixed and filtered through a dry filter; 10 ml. of the filtrate is pipetted into a 100-ml. volumetric flask and solid NH_4F is added, until the colour is discharged. Then 25 ml. of NH_4CNS and 50 ml. of acetone is added, diluted to mark, mixed and filtered through a dry filter and measured photometrically with red filters.

LIME.—The determination of calcium is based nearly always on the precipitation of calcium as oxalate, and titration with $KMnO_4$. The method requires the removal of interfering iron and aluminium by ammonia. In presence of large amounts of ammonium salts calcium precipitates slowly and the results are also frequently low. For greater accuracy iron is first removed by electrolysis with mercury cathode. By means of tartaric and oxalic acid it is possible to mask the interference of iron and aluminium. The use of citric acid is not so profitable.

Procedure.—0·5 g. of the slag is decomposed as above, some HCl is added to dissolve $CaSO_4$. Tartaric and oxalic acids are added and the solution made ammoniacal, boiled and cooled under the tap. Calcium oxalate is filtered and calcium determined with $KMnO_4$ in the usual manner.

MAGNESIA.—Magnesia is usually determined after the removing of iron, aluminium and calcium as phosphate. The use of mercury cathode for removing of interfering elements is profitable. In addition, the use of oxine makes the volumetric determination of magnesium possible.

Procedure.—1 g. of the slag is decomposed as above; NH_4Cl is added and the boiling solution neutralised with ammonia; NH_4-oxalate and NH_4-sulphide are added in a slight excess to precipitate Cu, Fe, Al, Zn, Ni, Co and Ca. The solution is transferred into a 200-ml. volumetric flask, cooled, diluted to mark, mixed and filtered through a dry filter; 100 ml. of filtrate is pipetted, oxidised with H_2O_2 and filtered, if necessary. Ammonia is added and magnesium is precipitated with 5% oxine in alcohol. The precipitate is filtered, ignited with oxalic acid to MgO. Magnesium may be also determined volumetrically with $KBrO_3$.

Alkalies

The determination of sodium and potassium in slags is sometimes required. The use of mercury cathode and the removing of the bulk of ammonium salts by means of aqua regia simplifies considerably the usual method. Before and after electrolysis the excess of H_2SO_4 is eliminated by heating the solution to dryness. Large excesses of different reagents must be avoided by using diluted solutions and litmus as indicator. Otherwise potassium and sodium are determined according to the general methods.

SULPHUR.—Sulphur is determined accurately with $BaCl_2$ as usual. The slag is decomposed with Na_2O_2. The solution of the melt is filtered with suction on Buchner funnel, and the filtrate is acidified with HCl, or the iron may be immediately reduced in HCl solution with hydroxylamine hydrochloride. More rapid is the combustion of 0·25 g. of the slag in oxygen at 1,200° C. by using 1 g. of tin as accelerator. SO_2 is absorbed with 4% H_2O_2 and titrated with n/10 NaOH methyl red as indicator.

MANGANESE.—The method of Proctor-Smith is suitable for determination of manganese in slags. Manganese is oxidized with $(NH_4)_2S_2O_8$ in presence of $AgNO_3$ as catalytic agent. The cooled solution is titrated with Na-arsenite. When preferred, NaCl may be added before titration. For the detection of minute amounts the removing of iron by ether is recommended.

PHOSPHORUS.—For the determination of phosphorus the slag is decomposed as above, and the hot solution is made ammoniacal and boiled. The precipitate is filtered, washed with hot water and dissolved with HNO_3. Phosphorus is precipitated with NH_4-molybdate and titrated with alkali solution as usual.

TITANIUM.—The determination of titanium in slag does not meet any difficulties, if the slag is decomposed as above and if iron and other interfering elements are removed electrolytically by mercury cathode. Titanium is then precipitated together with aluminium by ammonia and may be determined in an acid sulphate solution either colorimetrically by H_2O_2 or by precipitation with cupferron. The precipitate is filtered, washed and ignited to TiO_2.

Analysis of Mattes

COPPER.—Copper in mattes is rapidly determined either by electrolysis in presence of iron or by iodide method, modified by Park[11].

Procedure.—0·5 g. of the mattes is decomposed in a 300-ml. Erlenmeyer flask with a few drops of bromine and 20 ml. of HNO_3 1 : 4. When the reaction is over, the solution is boiled, heated with 5 ml. of H_2SO_4 1 : 1 over a free flame to fumes and cooled. The residue is taken up with 50 ml. water and boiled. The solution is made slightly ammoniacal and cooled. Solid KHF is added, until pure blue colour appears; 1–2 g. of KJ is added and the solution is titrated with n/10 $Na_2S_2O_3$ as usual. If only copper is to be determined, some NH_4CNS is added to make the endpoint sharper.

IRON.—In addition to the $KMnO_4$- and $TiCl_3$-method iron may be also determined iodometrically as described by Lang and Reifer.[6]

Procedure.—When copper is titrated as described above, 10–15 ml. of 5 n H_2SO_4, 3 g. of boric acid and about 1 g. of KCNS are added and iron is then titrated with n/10 $Na_2S_2O_3$.

ZINC.—If nickel and cobalt are absent, zinc may be determined easily with iodide method. After copper or iron is titrated, the solution is boiled, filtered and washed with hot water. The filtrate is cooled, the blue colour is removed exactly with n/10 $Na_2S_2O_3$ and zinc is determined as described in connection with the slag by $K_3Fe(CN)_6$ and $Na_2S_2O_3$.

COBALT.—Cobalt is rapidly determined as described in connection with the slag. Large amounts of nickel interfere, however, and account for its green colour. In this case it is necessary to extract cobalt with amylalcohol-ether mixture.

Procedure.—0·5 g. of the slag is decomposed as above. Copper is precipitated in slightly acid boiling solution with 10 ml. of 10% hydroxylamine hydrochloride and 20% NH_4CNS avoiding an excess. To 10 ml. of filtrate in separatory funnel 2–3 g. of NH_4F and 25 ml. of 20%

9 Tomula, E., *Z.f. anal. Chem.*, **83** (1931), 8.6.
10 Ringbom, A., Unpublished work.
11 Park, B., *Ind. Eng. Chem. Anal. Ed.*, **3**, 77 (1931).

NH_4CNS are added. The solution is extracted several times with 20 ml. of amylalcohol and 80 ml. of ether, and cobalt is determined in extracts colorimetrically with red filters.

NICKEL.—Nickel in mattes is easily separated with dimethylglyoxime and may then be determined electrolytically, gravimetrically, volumetrically according to Mironoff[12] and Lindström[13] or colorimetrically according to the author.[14]

Volumetric Method

Procedure.—0·5 g. of the slag is decomposed as above. Nickel is then precipitated in a slightly ammoniacal tartrate solution with dimethylglyoxime as usual. The washed precipitate is rinsed with water into the beaker 25 ml. of 20% ferric ammonium sulphate and 5 ml. of H_2SO_4 1 : 1 are added and boiled at least 10 minutes. The solution is diluted with cold water to 300 ml. and titrated with n/10 $KMnO_4$. 1 ml. n/10 $KMnO_4$ = 0·738 mg. Ni.

Colorimetric Methods

Procedure.—(*a*) The nickel-glyoxime-precipitate containing about 50 mg. or more of nickel is decomposed with HNO_3 and heated with 10 ml. of H_2SO_4 1 : 1 to fumes. The residue is dissolved with water diluted to 100 ml. and measured with red filters.

(*b*) 0·5 g. of thematte is decomposed as above ; 10 ml. of H_3PO_4 1 : 7 and 30 ml. of solution (50 g. of thio-urea, 1,000 ml. of water and 500 ml. of H_2SO_4 1 : 1) are added. The solutionis diluted to 100 ml. and measured. Instead of thio-urea and phosphoric acid, zinc may also be used.

SULPHUR.—Sulphur may be determined in mattes either according to Lunge or with Na_2O_2 as described in connection with the slag. The most rapid method, however, is the combustion in oxygen. The results are satisfactory, if they are corrected by means of a sample the composition of which is known.

OXYGEN.—In mattes there is usually oxygen due to the magnetite. It may be easily determined by heating the mattes in a current of oxygen-free hydrogen at 800°–900°C. The water is absorbed with P_2O_5-tube which is first saturated with H_2S.

Summary

The different slags and mattes vary so widely in their chemical composition that it is difficult to lay down precise and detailed methods for treating all slags and mattes. However, these general methods may be used in the majority of cases and these are not only restricted to the slags and mattes, but may be applied with appropriate modifications to silicate, rocks, tailings, concentrates, etc. The above methods have been found very satisfactory as regards both speed and accuracy and they have been used several years. The time required for determination of SiO_2, Cu, Fe, Al, Zn, Ni, Co, CaO, MgO or S varies from 10–60 minutes.

Acknowledgments

The author is especially indebted to Professor A. Ringbom and Mr. T. Lindström for their kind permission to publish details of their unpublished works and also to many others for their laboratory works. Thanks are also due to Outokumpu Oy for permission to publish this paper.

12 Mironoff, J., *Bull. Soc. Chem., Belg.*, **45**, 1, 1936.
13 Lindström, T., Unpublished work.
14 Kinnunen, J., *Metall. und Erz*, 1944, **41**, 158.

Modern Applications of High Temperature Alloys

A COURSE of six lectures on the above subject has been arranged by the Department of Metallurgy at the Sir John Cass Technical Institute, Jewry Street, Aldgate, E.C.3. The lectures, which will be given on Thursday evenings at 6-15 p.m. commencing on January 22, will be given by Mr. C. G. Conway, B.Sc. As will be noted in the following syllabus, the subject is considered comprehensively. The first lecture is of an introductory character and will be concerned with an outline of historical development of high temperature alloys to 1939 ; important factors in design—significance of creep, rupture, fatigue and physical properties—oxidation and corrosion ; survey of special testing technique—utilisation of data in design. The second will deal with uses and properties of ferritic steels used as high temperature materials and will be concerned with the work done on carbon and carbon-molybdenum steels—uses in steam turbine practice—survey of chromium, chromium-molybdenum and nickel-chromium-molybdenum ferritic steels—utilisation of data in practice. Uses and properties of austenitic steels will be considered in the third lecture in which physical properties and creep data on austenitic steels will be surveyed and the recent researches on improved austenitic steels summarised. The fourth lecture will deal with miscellaneous elevated temperature materials and will include cast irons, bronzes, nickel-copper alloys and aluminium alloys used for elevated temperature applications. Special problems in the gas turbine with their present day solution will be considered in the next, and the final lecture will survey the properties of new alloys developed in United States and the United Kingdom for gas turbine applications.

Each lecture will last about one hour. The fee for the course is 10s. Application for enrolment should be made to the Principal prior to the opening date, if possible.

Nichrome

IN dealing with developments in alloy and special steels of page 75 of the last issue of this journal reference is made to a nickel base alloy of the Nichrome type, but we omitted to mention that the word "Nichrome" is the registered Trade Mark of British Driver Harris Co. Ltd., Manchester, 15. Although this is known by many readers, others may not bo so familiar with the association of the term.

New Selling Prices for Tin Metal

THE Ministry of Supply announces that on and from December 17, 1947, the price of tin metal of minimum 99%, up to under 99·75% tin content, is increased from £437 to £510 per ton (f.o.b. U.K. port or delivered U.K. consumers' works). Other grades are varied as follows :

Refined Tin 99·75% minimum, from £438 10s. to £513 10s. ; Refined Tin 99·9% minimum in 28 lb. ingots, from £443 to £518 ; Grain Bar Tin, from £457 to £530 ; Granulated Tin, from £462 to £535.

The restrictions on the granting of licences to U.K. consumers which were in force for November and first half December 1947 are now withdrawn, and applications for such licences will, until further notice, be granted in full at the new prices now announced. Enquiries should be addressed to the Directorate of Non-Ferrous Metals, 20, Albert Street, Rugby. (Rugby 2131.)

MICROANALYSIS
CHEMICAL AND PHYSICAL METHODS
APPARATUS · METALLURGICAL APPLICATIONS · TECHNIQUE

PROBABLY the most important news of recent months for the analytical chemist is the announcement, in the October issue of "Analytical Chemistry," of the Fisher Award in Analytical Chemistry, for the encouragement and recognition of outstanding contributions to the science of analytical chemistry, pure or applied, carried out in the United States or Canada. For chemists in this country this seems, at first sight, by reason of its territorial limitation, to be merely another news item. And it is not the value of the award, which is substantial, that is of importance, but the implications of the award make us wish that we were as silver-tongued as the writer of the editorial in "Analytical Chemistry" for March, to which the award was, at least in part an answer. Here we have one of the first concrete signs that someone recognises the importance of fundamental analytical research sufficiently to be prepared to pay for it in hard cash. It is probably true to say that the recipients-to-be of the award would feel the honour just as much were it to consist of a medallion only, without the dollars. The latter, however, are some measure of the regard in which a large industrial organisation holds the profession of analytical chemistry. We sincerely hope that it will not be too long before someone in this country is sufficiently appreciative of the action of the President of the Fisher Scientific Company to make some attempt to flatter him by imitation.

A Review of Micro Methods for the Determination of Oxygen in Organic Compounds*

By C. E. Spooner

It is intended in this review to deal with the developments which have taken place in the method for the direct determination of oxygen with particular reference to micro scale. It is not claimed to be comprehensive, and no experimental details will be given, although most of the original references will be quoted which can be consulted if required. The paper follows from the extensive search of the literature which was made by the author in developing a macro-method for the direct determination of oxygen in coal,[1] and the author has himself had no experience of the determination of oxygen on the micro-scale.

THE usual method for the determination of oxygen in organic compounds is to determine all the other constituents and subtract the total from 100. It will be realised that this method takes a long time, and all the errors in the other constituents accumulate in the figure given for oxygen. The reason for this is the experimental difficulties in a direct determination of oxygen. It is not due to lack of methods as over 100 papers have been published on this subject. They can be divided into two groups: (1) Oxidation methods; and (2) reduction methods.

Oxidation Methods

Oxidising agents.—The earliest method was put forward by Gay-Lussac and Thenard[2] in 1811. The method was to oxidise the substance with potassium chlorate and analyse the products. The amount of potassium chlorate used was determined and the oxygen supplied by this calculated. The difference between this figure and the total oxygen in the products was taken as the oxygen content of the organic substance. Similar methods using solid oxidising agents have been put forward using mercuric sulphate,[3] mercuric oxide,[4] litharge[5] and cupric oxide.[6-11]

Other of the earlier methods used wet oxidising agents such as silver iodate in concentrated sulphuric acid[12] and a mixture of chromic and sulphuric acids.[13] One such method was put forward by Strebinger, who used

*Paper read at the May Meeting, in Sheffield, of the Microchemistry Group of the Society of Public Analysts and Other Analytical Chemists.

1 Spooner, C. E., *Fuel*, 1947, **26**, 15–23.
2 Gay Lussac, J. L., and Thenard, L. J., *Ann. Chem.*, 1,811, **79**, 60–62.
3 Persoz, J., *Ann. Chim. Phys.* (4), 1,840, **75**, 5–24.
4 Mitscherlich, A., *Ber.*, 1873, **6**, 1,000–2; 1874, **7**, 1,527; *Z. anal. Chem.*, 1876, **15**, 371–417.
5 Maumene, E. J., *Compt. rend.*, 1862, **55**, 432–435.
6 Baumhauer, E. H. Von, *Ann.*, 1854, **90**, 228–231; *Z. anal. Chem.*, 1866, **5**, 141–152.
7 Boswell, M. C., *J. Amer. Chem. Soc.*, 1914, **36**, 127–132.
8 Stromeyer, ..., *Ann.*, 1861, **117**, 247–260.
9 Prout quoted in ref. 8 above.
10 Porret, *Schweigg Journal*, **17**, 297.
11 Trautmann, W., *Z. Physik. Chem. Unterricht.*, 1940, **53**, 142–144.
12 Ladenburg, A., *Ann.*, 1865, **135**, 1–24.
13 Phelps, I. K., *Am. J. Sci.*, 1897, **154**, 379–382.

potassium iodate and sulphuric acid.[14] He heated the mixture until all the iodine had been given off, then the excess iodate was titrated with thiosulphate. This method was adapted to the micro-scale by Stanek and Nemes,[15] and it gave good results if the compound contained only carbon, hydrogen, oxygen, sulphur and chlorine, but when nitrogen was present only poor results were obtained.

The disadvantages of these methods using oxidising agents are: (1) The number of weighings which are necessary and the complicated calculation to obtain the result; (2) the oxygen content is only obtained indirectly as the difference between the total oxygen in the product and that supplied by the oxidising agent; (3) errors are likely to be introduced due to the formation of some carbon monoxide; (4) the carbon and hydrogen contents of the substance must be determined in calculating the oxygen content; (5) the oxidising agent must be pure and the disadvantages peculiar to each oxidising agent must be overcome.

Oxidation with a known volume of gaseous oxygen.—This is the most satisfactory of the oxidation methods and briefly consists of combustion of the organic compound in the usual carbon-hydrogen combustion train but with the oxygen circulating in a closed system. The first satisfactory technique was put forward by Glockler and Roberts[16] in 1928, who gave the technique for a semi-micro method using 30–35 mg. The oxygen was obtained by heating potassium permanganate and the combustion train contained platinised asbestos as a catalyst for the oxidation. The combustion took 45 minutes and the type of result obtained was that benzoic acid (26·25% theoretical) gave a figure 0·7% low. Since this original specification there have been a number of modified techniques put forward.[17–24]

This technique was studied in detail on the micro-scale by Kirner in 1934,[25] and the conclusions he reached were as follows:—

1. The temperature must be kept as near constant as possible, and in his technique he enclosed the whole of his apparatus in a constant temperature bath. Failing this, the appropriate corrections for change in temperature and pressure must be made when measuring the volume of oxygen used in the combustion.

2. A blank determination must be made on the apparatus to ascertain the rate of diffusion of oxygen through the rubber connections.

3. The carbon and hydrogen content of the organic compound must be determined correctly as any error in the carbon content is multiplied 2·7 times in the oxygen figure, and any error in the hydrogen content is multiplied 8·0 times.

4. If the compound contains sulphur, then this is oxidised to sulphur trioxide which is absorbed as silver sulphate if silver wire is present in the combustion tube.

5. If nitrogen is present in the compound then this is liberated either as gaseous nitrogen or as nitrogen di-oxide. The ratio of these two products depends on the way in which the nitrogen is linked in the organic compound; amines and amides liberate 26% of the nitrogen as nitrogen peroxide and 74% as free nitrogen; nitriles, nitro and heterocyclic nitrogen compounds liberate 59% nitrogen peroxide and 41% free nitrogen.

Among the industrial organic compounds, coal liberates 59% nitrogen peroxide and 41% free nitrogen.

The disadvantages of this method are the need for an accurate figure for carbon and hydrogen content, and the necessity for the separate determination of sulphur and nitrogen if these are present. In addition, when nitrogen is present some knowledge of the way in which it is linked must be ascertained.

When all these disadvantages have been overcome, the oxygen can only be determined to within 0·5%.

Reduction Methods

These methods are the most satisfactory means of determining the oxygen content as only the oxygen which is present in the organic compound takes part in the determination. They can be divided into two main classes: (1) Hydrogenation methods; and (2) Reduction over hot carbon.

1. *Hydrogenation methods.*—This technique was first suggested by Wanklyn and Frank in 1863,[26] but the experimental details were first given by Ter Meulen in 1922,[27] and the method is usually associated with his name. The essentials of the method are: (1) Vaporise the organic compound in a stream of hydrogen; (2) crack the vapour at a high temperature; and (3) hydrogenate the cracked product over nickel at 300°–400° C. when all the oxygen is converted into water.

At first Ter Meulen only used a hydrogenating catalyst, but he later added a cracking catalyst consisting of either asbestos or platinised asbestos.[28] Since his original specification a number of papers have dealt with this subject,[29–42] and the modifications in conditions and catalysts are too numerous to mention in detail. The various suggestions for catalysts are as follows:—

Cracking catalyst—asbestos, platinised asbestos, platinised quartz, nickel spiral and nickel chromite.

Hydrogenating catalyst—nickel and asbestos, nickel powder, thoria-activated nickel, nickel and pumice, and nickel chromite.

The two most outstanding modifications were by Russell and co-workers[43] who first showed that thoria promoted the nickel as a hydrogenating catalyst, and since their specification thoria-promoted nickel has invariably been used as the hydrogenating catalyst. The other noteworthy contribution was by Goodlow and

14 Strebinger, R., *Z. anal. Chem.*, 1919, **58**, 97–114.
15 Stanek, V., and Nemes, T., *Chem. Ztg.*, 1932, **56**, 285–287; *Z. anal. Chem.*, 1935, **99**, 244–260.
16 Glockler, G., and Roberts, L. D., *J. Amer. Chem. Soc.*, 1928, **50**, 828–831.
17 Dolch, M., *Proc. Int. Conf. Bit. Coal. 2nd Conf.*, 1929, 425–447.
18 Dolch, M., and Will, H., *Brenn. Chem.*, 1931, **12**, 141–146.
19 Beek, F., van, and Waard, S. de, *Brenn. Chem.*, 1931, **12**, 402–403.
20 Schuster, F., *Brenn. Chem.*, 1931, **12**, 403.
21 Dumke, W. A., *Quart. Colo. School Mines*, 1931, **26**, No. 2, 5–21.
22 Stock, A., Lux, H., and Rayner, J. W. R., *Z. anorg. allgem. Chem.*, 1931, **198**, 58–63.
23 Hellman, S., *Feuerungstechnik*, 1933, **21**, 136–138.
24 Dolch, M., and Will, H., *Brenn. Chem.*, 1931, **12**, 166–169.
25 Kirner, W. R., *Ind. Eng. Chem., An. Ed.*, 1934, **6**, 358–363; 1935, **7**, 363–365; 1935, **7**, 366–368; 1936, **8**, 57–61.
26 Wanklyn, J., and Frank, *Phil. Mag.* (4), 1863, **26**, 554.
27 Meulen, H., ter., *Chem. Weekblad.*, 1922, **19**, 191–192.
28 Meulen, H., ter., *Rec. Trav. Chim.*, 1924, **43**, 899–904; *Chem. Weekblad.*, 1926, **23**, 348–349.
29 Meulen, H., ter, and Heslinga, J., *Nieu Methoden voor Flemintairacly se Meneme Delft* (1926) (translated in French and German).
30 Afanasev, B. N., *Zavodskaya Lab.*, 1937, **6**, 551–552.
31 Blacher, C., *Feuerungstechnik*, 1925, **13**, 69–70, 84–86, 95–98, 126–127, 148–152.
32 Brender, A., and Brandis, G. A., *Het. Gas.*, 1932, **52**, 57.
33 Brender, A., and Brandis, G. A., *Z. Oster Ver. Gas., Wasserfach*, 1934, **74**, 181–183.
34 Inaba, T., and Abe, Y., *J. Soc. Chem. Ind., Japan*, 1936, **39**. Suppl. binding, 91.
35 Meulen, H. Ter, *Brenn. Chem.*, 1931, **12**, 401–402.
36 Morikawa, K., Kimoto, T., and Abe, R., *Bull. Chem. Soc. Japan*, 1941 **16**, 229–232.
37 Schuster, F., *Gas u. Wasserfach*, 1930, **73**, 549–551.
38 Schuster, F., *Brenn. Chem.*, 1931, **12**, 403.
39 Schuster, F., *Het. Gas*, 1932, **52**, 56–57.
40 Untersaucher, J., and Burger, K., *Ber.*, 1938, **71B**, 429–443.
41 Meulen, H., ter., *Brenn. Chem.*, 1931, **12**, 401–402.
42 Will, H., *Brenn. Chem.*, 1931, **12**, 423–424.
43 Marks, M. E., *Ind. Eng. Chem., An. Ed.*, 1935, **7**, 102–103; Russell, W. W., and Fulton, J. W., *ibid.*, 1933, **5**, 384–386; Russell, W. W., and Marks, M.E., *ibid.*, 1934, **6**, 381–382, 1936, **8**, 453–455.

Frazer[44] who suggested that nickel chromite should be used for both catalysts; as a cracking catalyst at a red heat when it absorbs all the sulphur and so prevents this element from poisoning the hydrogenating catalyst; as a hydrogenating catalyst at a temperature of 300–400 when it has the advantage of producing only free nitrogen if this is present in the organic compound.

This hydrogenating method was also investigated in detail on the micro scale by Kirner,[26] who used nickel at 1,000° C. as a cracking catalyst and thoria-promoted nickel at 350° C. as the hydrogenating catalyst. He found that if only carbon, hydrogen and oxygen were present in the organic compound, then the oxygen could be determined to within 0·1%, but he obtained inexplicably low results for poly-hydroxy compounds such as sucrose. If the compound contained sulphur then this was converted to sulphuretted hydrogen which poisoned the hydrogenating catalyst if means were not adopted for removal of the sulphuretted hydrogen before it reached the hydrogenating catalyst. Various methods were suggested to accomplish this, such as the presence of calcium oxide in the reaction tube between the two catalysts. If chlorine was present in the organic compound then this was finally liberated as hydrochloric acid which would be absorbed with the water unless precautions were taken against this. One method which has been suggested to overcome this is to have a double absorber, the first bowl containing silver nitrate solution and the second bowl a suitable desiccating agent. The hydrochloric acid is absorbed in the silver nitrate solution and the amount produced can be determined by back titration of the excess silver nitrate. The gain in weight of the double absorber is equal to the hydrochloric acid and water produced in the reaction. If nitrogen is present in the organic compound and nickel or thoria-promoted nickel is used as the hydrogenating catalyst, the nitrogen is liberated as ammonia and similar precautions must be taken.

The water produced is usually determined gravimetrically by absorption in a suitable desiccating agent such as calcium chloride, anhydrone or phosphorus pentoxide. The water has, however, been determined by other methods, as Linker and Wirth absorbed the water in naphthyldichloro phosphine, which produced an equivalent amount of hydrochloric acid when it reacted with water. Lacourt used cinnamyl chloride which reacts likewise.

The advantage of hydrogenation methods is that the oxygen is determined directly and there is no need to determine the carbon and hydrogen contents of the compound. If chlorine is present, however, it is usually necessary to determine this. The disadvantages of the method are:—

1. The complications which are introduced when elements other than carbon, hydrogen and oxygen are present. In the case of coal, the ash content adds to the complication as some of the oxygen in the ash is hydrogenated to water.

2. The poisoning of the catalysts by sulphur and chlorine.

3. The large amount of carbon which is deposited on the cracking catalysts necessitating a burning off after each determination.

4. The poor conversion factor—namely, 0·88, for converting the water to oxygen.

44 Gaudoe, P., and Frazer, J. C. W., *Ind. Eng. Chem., An. Ed.*, 1937, **9**, 223–225.

Reduction over hot carbon.—The basis of this method is to decompose the compound and sweep the products by means of a stream of nitrogen over carbon at a high temperature, when the oxygen is converted into carbon monoxide. The method was first suggested by Boswell in 1913,[45] who used hydrogen as the carrier gas and converted the oxygen into water, carbon dioxide and carbon monoxide, and a separate determination of each product was necessary. The method in its present form was first developed by Schutze in 1939,[46] who put forward a semi-micro method using nitrogen as the carrier gas. He volatilised the compound and passed it over carbon at 1,000° C. The carbon monoxide formed was determined by passing it over iodine pentoxide which oxidised it to carbon dioxide, being itself reduced to iodine. The determination can be made either by means of the carbon dioxide produced or the iodine liberated, and Schutze reported an accuracy of ± 0·2%, each determination taking 1 hour for completion. The method was adapted to the micro-scale by Zimmerman,[47] who used samples of 5 mg. and obtained the oxygen correct to 1% relative value even when the substance contained carbon, hydrogen, nitrogen, sulphur and chlorine. The disadvantage of Zimmerman's technique was the large blanks (100 gamma.) on his apparatus due to the air entrapped with the sample when it was introduced. The method was employed by Unterzaucher,[48] who claims to have reduced the blank to zero. He accomplished this by means of a system of valves to sweep out any air which was introduced with the sample. He also increased the temperature of the carbon to 1,120° C., showing that this was necessary on theoretical grounds to enable the reactions to proceed to completion. The carbon monoxide formed was determined by oxidation with iodine pentoxide, the iodine liberated being absorbed in caustic soda, oxidised by means of bromine to iodate treated with potassium iodide solution, acidified and titrated with $\frac{N}{50}$ thiosulphate solution. Under these conditions 1 ml. of $\frac{N}{50}$ thiosulphate is equivalent to 0·1335 m.g. of oxygen. Unterzaucher obtained an accuracy of 0·1% and his determination was completed in 30–40 minutes.

The present author[1] when investigating this method on the macro-scale increased the temperature of the carbon still further up to 1,200° C., and he considers that this is necessary to complete the reactions under the dynamic equilibrium which exists in the reaction tube. He also determined the carbon monoxide formed by adding air to the decomposition gases and passing the mixture over hopcalite, a mixture of inorganic oxides which oxidises carbon monoxide to carbon dioxide at room temperature without any effect upon hydrocarbons.

The disadvantage of this carbon reduction method is the high temperature which is necessary. This is above the safe limit of a nichrome wound furnace, but is easily attained by one wound with platinum wire or one heated with carborundum heating elements. Attempts made by the author to reduce the temperature by using carbon activated with sodium oxide proved unsatisfactory. The activated carbon was originally very

45 Boswell, M. C., *J. Am. Chem. Soc.*, 1913, **35**, 284–290.

46 Schutze, M., *Naturwissenschaften*, 1939, **27**, 822; *Z. anal. Chem.*, 1939, **118**, 241–245, 245–258.

47 Zimmermann, W., *Z. anal. Chem.*, 1939, **118**, 258–263.

48 Unterzaucher, J., *Ber.*, 1940, **73B**, 391–404.

satisfactory and completed the reduction to carbon monoxide at a temperature of 800° C., but after the first determination it was blanketed by carbon deposited from the organic compound and ceased to function satisfactorily. The advantages of the method are:

1. The simple apparatus required apart from the high-temperature furnace.
2. The lack of any complicated calculation.
3. The fact that there is no catalyst to poison.
4. Carbon which is deposited during the decomposition merely increases the amount of reactive substance present.
5. The conversion factor is very good, being 0·3636.

It is most probable that future developments of methods for the direct determination of oxygen will be based on the carbon reduction method, as this is the simplest to operate and the most reliable.

An Apparatus for the Detection and Estimation of Gases Evolved in the Analysis of Carbonates, Oxalates, Sulphides, Sulphites, etc.

By J. G. Reynolds

A SIMPLE, but ingenious apparatus for the detection of evolved gases has been suggested by O. F. Stafford.[1] This apparatus is illustrated in Fig. 1 and consists of a tube with a capillary point for containing the reagent—e.g., calcium or barium hydroxide in the case of carbonates and oxalates. He claimed that "the introduction of the point of the capillary into any space where carbon dioxide may be, and the simultaneous aspiration of a few bubbles of gas, from that space into the reagent in the capillary, will produce the familiar carbonate precipitate with great certainty and delicacy."

This apparatus suffers from some disadvantages. The capillary point must be fine enough to permit a small quantity of reagent to remain in the tube without dripping on to the test material, but not too fine to prevent the passage of gas bubbles upward through the reagent, when gentle suction is applied to the open end of the reagent tube. In view of this, a modification has been devised[2] as illustrated in Fig. 2. This consists of a capillary tube (rather shorter and more robust than in Fig. 1) sealed into a small bulb, into which the reagent is introduced through a hole (2–3 mms. diameter) by means of a capillary pipette. The level of the reagent is slightly above the tip of the capillary. This entire reagent tube fits loosely into the reaction tube and is held in place by a rubber sleeve. In practice the reaction is initiated and the reagent tube then forced through the rubber sleeve, already in position round the top of the reaction tube. As the reagent tube is introduced further into the reaction tube, evolved gases, which cannot escape from the system owing to the tight rubber sleeve, are forced through the reagent. Gas pressure forces the reagent up the capillary until gravity causes it to sink back into the bulb, when the operation is repeated. The disadvantage of this apparatus (Fig. 2) lies in the fact that the reaction must be initiated before the reaction tube can be inserted. A tight rubber sleeve is necessary to prevent loss of gas and this sleeve is often the cause of difficulty in inserting the reagent tube. The use of a lubricant between the sleeve and the reagent tube is undesirable.

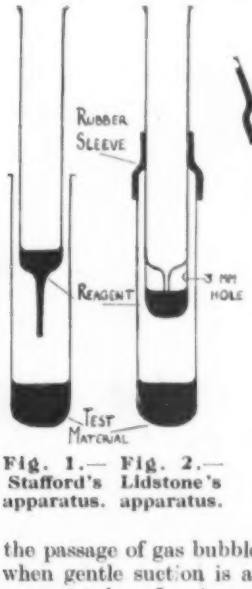


Fig. 1.— Stafford's apparatus.

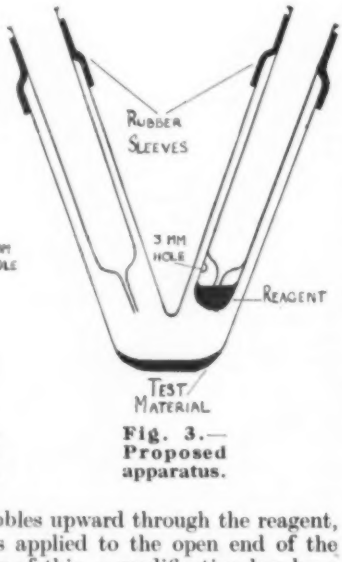


Fig. 2.— Lidstone's apparatus.

Fig. 3.— Proposed apparatus.

A further modification is therefore suggested and illustrated in Fig. 3. This consists of a reaction tube bent into a V and fitted with rubber sleeves at both open ends. The test material is introduced into the bend of the V and the reagent tube (illustrated in Fig. 2 and containing reagent) fitted into one arm of the V through the rubber sleeve. Through the other rubber sleeve is fitted an empty capillary (similar to the reagent tube shown in Fig. 1). The whole apparatus is now fairly rigid and robust and may be lightly clamped. Gentle aspiration is applied to the reagent tube and a small volume of reactant introduced into the other capillary tube. The reactant is quickly drawn down on to the test material and the gas evolved passes through the reagent.

The device may be made quantitative for minute quantities of carbonates, oxalates, sulphides and sulphites, the reagent after test being thoroughly washed out of the bulb and examined nephelometrically colorimetrically.

The author's thanks are due to "Shell" Refining and Marketing Co., Ltd., for permission to publish this note.

1 Stafford, O. F., *J. Am. Chem. Soc.*, **40**, 1, 622 (1918).
2 Lidstone, A. G. Unpublished.

Reviews of Current Literature

ALTHOUGH the scarcity of paper continued to retard the publication of technical books during 1947, there was an appreciable increase in the number which became available. Many of these are directly concerned with the fields embraced by this journal and it is proposed to review briefly a number of these, which have been published in this country, together with some published in the United States.

Metallurgical Materials and Manufacturing Processes

This book is not only intended for students of metallurgy but for all engineers and workers connected with the ferrous and non-ferrous industry who seek information on the materials which they constantly use. It deals with the more important principles of the metallurgy of the general engineering materials, their properties, treatment and manufacture in a lucid and useful manner. The data included is of a comprehensive character and will be of great assistance to those seeking information on many aspects, which would normally necessitate searching many volumes.

The early chapters are concerned with the manufacture of iron and steel, mechanical testing, physical examination of metals, industrial control and measurement of temperature, heat treatment and mechanical working of steel. Separate chapters deal with carbon and alloy steels, cast iron, and malleable cast iron, while later chapters discuss non-ferrous metals and alloys, welding galvanising, centrifugal casting, chilled castings, and powder metallurgy, with a final chapter on the uses of metals and alloys during the war period.

It will be seen from this brief reference to chapters that the scope of this book is quite comprehensive, in view of which it can be regarded as a useful handbook, but containing more information than is usual with reference handbooks. The book is admirably written and its 340 pages provides a mine of information which is readily explored by mean of a very complete index. It is well illustrated and produced and should prove a great help to all concerned with metals.

By V. N. Wood, A.M.I.Brit.F., with a Foreword by J. W. Gardom; published by Chapman and Hall Ltd., 37, Essex Street, London, W.C.2; price 25s. net.

Cost Ascertainment Methods for the Ironfounding Industry

The costing of work in many iron foundries has always been a problem and some form of standardisation has for long been necessary, if only to reduce the many anomalies that have existed between one foundry and another on this subject. In 1943, the Council of Ironfoundry Associations appointed a Committee "to investigate costing methods in the industry and to formulate a system of cost ascertainment based on uniform principles." The system finally evolved by the Committee has been approved by the Council and its adoption by the Industry is strongly advocated.

The details of the system are set out in a book which has recently been distributed to all Members of constituent commercial Associations of the Council. The introduction points out that the system is described in such detail that it can be applied by the majority of foundries; it can be expanded for ironfoundries requiring greater particularisation of information, and contracted by smaller or less complex foundries. The principles, purposes and benefits of cost ascertainment on a uniform basis are briefly reviewed in Chapter I, and Chapters II to VI describe and explain the system under the headings: Requirements for cost ascertainment; classification of expenses; departmentalisation and allocation of expenses; metal and melting costs; and absorption of expenses into the costs of work done.

The application of the scheme is demonstrated in Chapter VII. Reference is made to a Master Plan (Appendix I), which covers the departmentalisation of the ironfoundry and its ancillary departments, and the classification, apportionment and absorption of all expenses. Appendices II to V reproduce examples of the periodical cost analysis, the trading account, the job cost ascertainment sheet, and estimate sheets. The charts and the letterpress are bound ingeniously into a twin-folding board cover which makes cross-reference easy. This book supplies a long felt need.

Circulation was originally intended to be confined to the Members of constituent commercial Associations; but the Council has now decided that non-members may be supplied with copies at the price of £2 2s. per copy. Applications, which should be accompanied by a remittance, should be sent to the Secretary, The Council of Ironfoundry Associations, Derbyshire House, Belgrove Street, London, W.C.1.

Aluminium Alloy Castings

In comparison with iron, copper, lead and zinc, aluminium can be regarded as a new material, but, in the past 25 years, great progress has been made in its development and application. During the recent war especially, it was in great demand and production reached very high levels. When the war ceased, a slump in the production of this material was expected, but changes in application have increased peace-time uses and the production of aluminium alloy castings is gradually approaching the peak war-time production. The improved technique and trained personnel developed during the war are being applied in this progress, but there is a scarcity of technical books that are concerned specially with aluminium alloy castings, this book, therefore, fills a gap and is an important contribution in the aluminium field.

In this book, immediately following the introductory chapter, the unusual course is taken of considering the factors involved in quoting for aluminium castings and following with pattern and die-making necessary for their production. To deal with the subject in strict chronological order the preparation of the mould should be next considered, but a chapter on melting is interposed in which the general properties of aluminium alloys are given, since they have a bearing on the moulding materials required. Subsequent chapters deal with sand control; mould and sand casting; die-preparation and casting; fettling and repairing; heat-treatment; inspection and laboratory assistance; after-treatment; machining; corrosion; and a final chapter in which the author considers the future outlook. This work also includes much tabulated data and is well illustrated and indexed. An excellent feature of this book is the wide range of references given.

This book is both interesting and informative; the subject is presented in such a manner that it cannot fail to be of value, not only to those engaged in the production of aluminium castings and their finishing, but also to users and potential users of them. Of the

many chapters considered to be outstanding that dealing with inspection and laboratory assistance is worthy of careful study, but the book is quite comprehensive and is recommended to all concerned with the production and use of aluminium castings.

By E. Carrington, M.Sc.; published by Charles Griffin and Co. Ltd., 42, Drury Lane, London, W.C.2; price 25s. net.

Kirkstall Steel Bar Specifications

This is the fourth edition of what has often been regarded as the best book of its kind published in this country, and this edition, the first published since the war began, admirably maintains the high standard set by earlier issues, indeed, keeping in mind present conditions, many will agree that the publishers have surpassed their previous edition. Not only is it excellently produced, but it is a steel reference book of a high order and one which contains information invaluable to the steel user.

The book opens with descriptive notes from which the reader readily appreciates the historical background of the Kirkstall Forge Works and the justifiable pride which is shown in the quality and standard of products apparent over a period which dates from the 12th century to the present day. Originally concerned with the manufacture and working of iron, which was gradually discontinued as the firm began to specialise in the production of steel, the work is now concentrated on the manufacture of steel bars in a wide variety of grades and conditions, in the manufacture of heavy drop forgings, and in the production of finished components for heavy commercial motor vehicles.

Many of the 130 pages present data on case-hardening, carbon and alloys steels; Kirkstall and standard steel specifications occupy about 50 pages, and useful tables and other data, at the end of the book, take up 8 pages. A very informative section presents, in a very lucid manner, notes on the composition and structure of steel, controlled grain size, heat treatment, the effect of mass on heat treatment, alloying elements, selection of steel, forging and rolling temperatures, case-hardening, endurance of steels in service, machinability, welding, and modern metallurgical developments in relation to steel. In addition, there is a useful technical reference section. The size of the book—9 in. × 11¼ in.—enables a complete specification to occupy one page with advantage to the user.

Readers familiar with earlier editions will welcome the opportunity of having this new and more up-to-date edition, others, particularly designers and engineers will find the book of great value, quite apart from the many interesting features it contains. The printer, Henry Jenkinson Ltd., Leeds, is worthy of high praise.

Compiled by The Metallurgical Department and published by Kirkstall Forge Ltd., Kirkstall, Leeds, England; price 7s. 6d. net.

Talks on Steelmaking

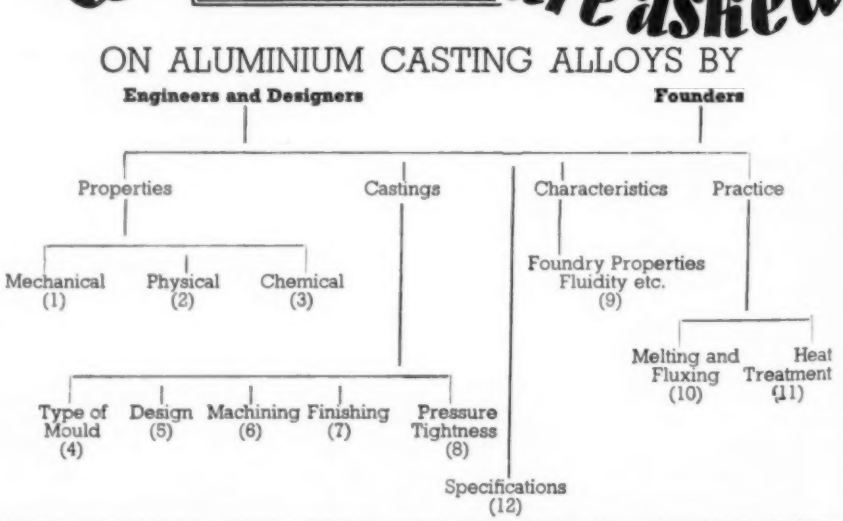
Those familiar with previous books by Harry Brearley need no introduction to the predominating characteristics of all his writings of which the most outstanding is forthrightness. This book, as Mr. Brearley says, is part suggestion, part history, part criticism, and essentially a biased point of view from which he has found steelmaking an attractive occupation. Practically

Introducing

Questions we are asked

ON ALUMINIUM CASTING ALLOYS BY

In our next twelve announcements we shall feature the type of questions which we answer daily for Engineers, Designers and Founders. It is the purpose of this Technical Association to give free and confidential advice to all who have problems connected with the use of Aluminium Casting Alloys. Every assistance is obtainable on request to the Association of Light Alloy Refiners.



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his whole life has been lived in and about steelworks, to which he owes most of his experience and education. His has been a hard schooling and steelworks are the background of his writings and it is not surprising that he has little room for those concerned with steelmaking who rely mainly on academic training, but his views have mellowed with time and he writes in such a charming manner that the reader cannot fail to derive pleasure from his books. This book is no exception, indeed, it can be regarded as his best.

It is doubtful whether anyone can make steelmaking so interesting and yet convey so much valuable information as is given in this book. The chapters are presented in the following order: beginnings, in which chemical analysis and microscopes are discussed; molten steel; melting processes; forging; testing steel; notch fractures; making specifications; clean steel; the job's the boss; complaints; remunerations; helpful comparison; and "scrapalurgy." This latter subject, which the author regards as an art, a science, a trade and an aspect of domestic economy, obviously refers to scrap; but consider the manner in which he writes about it. "All kinds of scrap have their own forms of picturesqueness; they are the returned remnants of an expeditionary force sent into the world to do something . . . A lifetime spent on a comprehensive waste recovery plant might qualify a patient observer to write a text-book for the ferrous trades on scrapology." None know better than the author that, quite apart from the economic aspect, the careful examination of scrap frequently supplies information for a story incorporating much of technical value.

There is a strong temptation to express some of the many truths which are expressed so admirably in this book. Considering the possibility of an ingot mould to produce tin-can surfaces, for instance, he writes, "It is easier to contrive experiments than to make them, but more delusive and not so interesting." Then of the melter who casts a spoon sample on a metal plate, into a flat piece which can easily be broken, to look at the fractured surface to observe its "teeth" . . . "What may be seen means more to the melter than the number of degrees centigrade which another observer might be writing in his book." But sufficient has been said to indicate the words he uses to clothe and present a life-time of experience and knowledge on a subject which becomes fascinating under the influence his book has on the reader.

The author suggests that the chapters may be read in any sequence; that they are addressed sometimes to the man and sometimes to the boy, but, whichever way they are read and whether by man or boy concerned with steelworks, the book will prove to be of such a character that the reader will quickly become absorbed in it. Because of paper difficulties it has been published in the United States and it is exceedingly well illustrated and produced.

By Harry Brearley; published by the American Society of Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.; price $3.50 post paid.

Induction Heating
Fundamental Principles and Applications

In recent years the advantages of induction heating have greatly increased its industrial applications. Formerly the only large-scale application was in the melting of metals, to-day it is used for hardening steel, brazing, soldering, forging, annealing, normalising, stress-relieving, paint-drying, etc., and this book describes the uses and possibilities of this form of heating.

Induction heating is the transfer of energy from an alternating magnetic field to a particular piece of metal, or section of that piece; the metal may be ferrous or non-ferrous and the heating takes place as a result of secondary eddy currents being forced to circulate on the surface of the metal itself when it is surrounded by a high-frequency current-carrying conductor. With this method of heating, distortion is held to a minimum because only the surface of the component under treatment is heated to critical temperatures. Absence of scaling, spalling, grinding cracks and of relief of hardening stresses during subsequent machining or grinding, and in many cases the obviating of pre-annealing, all result from the unusual speed of heating and uniformity of structure obtained.

The possibilities of this form of heating are very considerable and this book indicates the applications for which the method is suitable and economic, and will assist users and potential users in applying the method to the best advantage. The book is well written and illustrated and its 146 pages are full of information on the subject.

By "Heat-Treater," published by Chapman and Hall Ltd., 37, Essex Street, London, W.C.2; price 10s. 6d. net.

The Specification of Meehanite Steel

It is generally known that no one type of cast iron is suitable for all types of service conditions and this applies to Meehanite castings produced by controlled processes as to other iron castings. To assist designers, engineers and all concerning with the purchase of iron castings, to choose the type of Meehanite which will give the best service under the stresses imposed upon it, a very useful booklet was published some years ago. A new edition is now available which has been expanded to 48 pages and includes the results of more recent research.

Of particular interest is the bending and surface stress factors for various types of Meehanite metal. Useful articles included deal with the type of metal for heat resistance purposes; the effect of elevated temperatures on the strength properties; the effect of section thickness variation on physical properties; and the metallurgy of Meehanite metal. The main sections of this new booklet deal with general engineering castings, heat-, wear- and corrosion-resisting castings, and includes considerable general information and valuable data. Thus it provides the designer and engineer with background information as well as reliable data to assist in making the most satisfactory use of the type of metal for a specific purpose.

Copies of this booklet are available on application to The International Meehanite Metal Co. Ltd., 66, Victoria Street, London, S.W.1.

Messrs. New Metals and Chemicals Limited, 16, Northumberland Avenue, London, W.C.2, have been appointed Sole Distributors for the United Kingdom for Calcium Metal produced by Messrs. Dominion Magnesium Ltd.

Staff Changes and Appointments

MR. WILLIAM B. BAXTER, the General Manager of Appleby-Frodingham Steel Company, has been appointed a Director of The United Steel Companies Limited. He was trained as an Engineer and first became associated with the steel industry in 1914. At that time he joined the Frodingham Iron and Steel Company, now the Appleby-Frodingham Steel Company, and after assisting the late Mr. James Henderson became Works Manager in 1927 and General Manager in 1939. He is a member of Council and Vice-President of the Iron and Steel Institute, a member of Council and Chairman of the Plant Engineering Division of the British Iron and Steel Research Association, and Chairman of the Lincolnshire Ironmasters' Association.

MR. CHRISTOPHER LEONARD GALE FAIRFIELD, M.A., A.M.I.E.E., A.M.I.Mech.E., Barrister at Law, has joined the Mullard Wireless Service Company Ltd. The applications of Mullard research and development work to industrial problems will be one of Mr. Fairfield's many activities, in which he will act as assistant to the directors of the Company in a technical capacity.

E. S. WADDINGTON, M.S.E., M.Inst.W., A.M.I.E. (s.a.), Associate I.E.E., of Philips Electrical Ltd., who is a Member of the Council of the Institute of Welding, has been elected Vice-chairman of the Finance Committee of that Institute.

MR. HARTLAND THOMAS, M.A., F.R.I.B.A., M.S.I.A., has joined the Council of Industrial Design to take charge of its Industrial Section which is directly concerned with offering a practical service to industry on design problems, and particularly with helping industries to set up their own Design Centres.

MR. E. S. MCCALLISTER, who for many years has been connected with the electrical industry, particularly with the development of electronic instruments, has been appointed to the electro-medical department of Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2.

MR. ASHLEY S. WARD, chairman of Rhos. W. Ward, Limited, and other associated companies, has relinquished the chairmanship of Lowmoor Best Yorkshire Iron, Limited, and MR. GEORGE WOOD (deputy chairman of Thos. W. Ward, Limited) has been appointed in his place. Other changes on the board of this company include the election of MR. C. L. FRY and MR. W. W. HICKMAN as directors, and the appointment of MR. FRY and MR. R. STUBBS as joint managing directors. The position of works manager has been taken over by MR. G. F. HANSON.

MR. W. LIONEL FRASER, C.M.G., has been elected a director of Tube Investments Limited, and appointed Deputy Chairman. SIR EDWARD CRANE has resigned from the Board, prior to his leaving the country for a visit to South Africa.

MR. G. P. ROBERTS, M.I.Mech.E., has been appointed technical director to Guy Motors Ltd. He has had a wide engineering experience and, since his return from the Army, has been the Company's research and development engineer.

MR. C. F. RUSSELL has been appointed Chairman of the Aero Piston Ring Company Limited, and Mr. L. H. MAIDMAN and Mr. L. P. GIBSON of Specialloid Limited have joined the Board of the Aero Piston Ring Co. Ltd.

MR. H. COUSINS, publicity manager of the British Aluminium Co. Ltd., will be retiring at the end of January 1948, at his own request, after completing 40 years' service with this Company. As a consequence, the publicity department will be combined with the present sales research and statistical section under MR. C. G. MCAULIFFE, as manager. The new department will be known as publicity and sales research department and commenced operation on January 1st, 1948. MR. E. G. FIELDING, who is responsible for the publicity work of the department has the title of publicity officer.

MR. A. HORNER has recently been appointed General Manager of Darlington and Simpson Rolling Mills Ltd. which specialises in the rolling of light special steel sections.

MR. F. A. MARTIN and MR. F. W. ROWE were elected chairman and vice-chairman, respectively, for the ensuing year, at the recent Annual Meeting of the British Steel Founders' Association. Messrs. Peat, Marwick, Mitchell and Co., were re-elected secretaries of the Association.

MR. A. E. TAYLOR, who has been connected with Edgar Allen and Co. Ltd. for 44 years, has retired. He represented this Company over a large area in the East Midlands, with Leicester as his headquarters. MR. GILBERT FRANCIS, second son of Mr. S. H. Francis, commercial manager of the Edgar Allen Steel Foundry, has been appointed to succeed him as representative in the East Midlands area.

MESSRS. HADFIELDS LTD., East Hecla Works, Sheffield, announce the appointment of four additional local directors: MR. D. DIGBY HAWLEY, M.C., manager of the sales department, who has been in the service of the Company since 1919; MR. ALEC JACK, B.Met., who was recently appointed London manager after a number of years in the metallurgical and sales departments; J. R. RAIT, D.Sc., Ph.D., A.R.T.C., the Company's research controller and MR. F. COUSANS, F.I.M., M.I.B.F. foundry controller. These appointments have effect as from January 1st, 1948.

Correction

On page 90, December 1947 issue, 4th line from bottom of 1st column, "Mr. W. H. Bill" should read MR. W. H. BALL.

Obituary

It is with regret that we announce the death, on December 9th, 1947, of Mr. F. R. Wix, a Director of The Phosphor Bronze Co. Ltd., Witton, Birmingham.

His son, Mr. G. F. Wix, who has acted as his understudy for the past two years, will take over Mr. Wix's activities on behalf of this Company.

Institute of Metals Elections

THE Council of the above Institute has elected Dr. C. H. Desch, F.R.S. and Lieut-Col. Sir John H. M. Greenly, K.C.M.G., C.B.E., M.A., to be Fellows for eminent service rendered to this Institute. In addition, elections to honorary membership, for distinguished services to the science and practice of non-ferrous metallurgy, include Professor P. A. J. Chevenard, Officer de la Legion d'Honneur, Memre de l'Institut de France; Professor Sir G. T. Taylor, M.A., D.Sc., D.C.L., F.R.S.; and John F. Thompson, Ph.D.